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# A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

HELEN E. HINES

## MATERIALS LABORATORY

**BEST AVAILABLE COPY**

AUGUST 1957

**Statement A**  
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WADC TECHNICAL REPORT 53-373

SUPPLEMENT 4

ASTIA DOCUMENT No. 131001

# A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

*HELEN E. HINES*

*MATERIALS LABORATORY*

*AUGUST 1957*

WRIGHT AIR DEVELOPMENT CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO



## FOREWORD

This report was prepared by Miss Helen E. Hines of the Technical Data Section, Technical Services Branch, Materials Laboratory, Directorate of Laboratories, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

The author is greatly indebted to Miss Jennie L. Buckler, Mrs. Bobbi R. Clinkscales and Major Henry I. Lipsie for their assistance and counsel in preparing this report.

Technical Reports prepared by Materials Laboratory project engineers (those having no contract number) and Materials Laboratory contractors during the period 1 July 1956 - 30 June 1957 are abstracted herein.

Abstracts for Technical Notes of general public interest and Technical Reports not previously included are given in this supplement.

This report has been released to the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. for sale to the general public. To order copies of this report, address inquiries to the Department of Commerce.

Military organizations and prime contractors of the Department of Defense engaged in research and development should direct inquiries to Astia Document Center, Knott Building, ATTN: TIC-SCP, Dayton 2, Ohio.

The general public is invited to submit requests for copies of technical reports and technical notes which are abstracted in this report to the Department of Commerce. Only submit requests for reports that state OTS release. Military organizations and prime contractors should submit their requests with the Astia document number to Astia.

## ABSTRACT

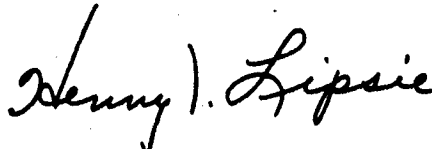
Two hundred and seventy (270) technical reports and technical notes written during the period 1 July 1956 - 30 June 1957 are abstracted. These reports cover the following areas of research: adhesives, metallurgy, analysis and measurement, biochemistry, textiles, petroleum products, plastics, packaging, protective treatments and rubber.

A contractor index, investigator index, and a numerical index of all the technical reports issued during the period March 1923 - June 1957 are provided.

## PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



HENRY I. LIPSIE  
Major, USAF  
Chief, Technical Services Branch  
Materials Laboratory

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ADHESIVES, STRUCTURAL

WADC TR 54-98 Pt 3  
ASTIA Document No. AD 97287  
OTS Release

September 1956

SUBJECT: INVESTIGATION AND DEVELOPMENT OF HIGH-TEMPERATURE STRUCTURAL ADHESIVES

INVESTIGATOR: Alfred S. Kidwell  
Kenneth L. McHugh

CONTRACT: AF 33(616)-2448

CONTRACTOR: The Connecticut Hard Rubber Company

ABSTRACT: Wide variations in the composition of the CHR-M-60 epoxy-modified DC-2103 silicone resin with Asbestine X filler, which has shown shear strength values as a metal-to-metal adhesive in excess of 1000 psi at 500°F, have been made and the results plotted to indicate composition areas yielding maximum shear strength values. Extended high-temperature aging tests have shown DC-803 silicone resin and epoxy-modified DC-803 to have better aging resistance than DC-2103 and epoxy-modified DC-2103, respectively. The DC-803 showed little loss in shear strength at 500°F after aging 1200 hours at 500°F on both aluminum panels and stainless steel panels (residual shear strength about 300 psi at 500°F). This resin also withstood 300 hours at 600°F on stainless steel and 200 hours on aluminum. The 200 percent epoxy-modified DC-803 lasted 800 hours at 500°F and 100 hours at 600°F (residual strength in each case about 350 psi at room temperature and 150 psi at 500°F).

A number of silicone-epoxy-phenolic resin blends were prepared, which showed shear strength values and high-temperature aging resistance slightly better than those for the epoxy-modified silicone resins.

Up to 20 percent of epoxy resin in epoxy-modified DC-2103 silicone resin was found to have relatively little effect on the change in shear strength values when tested over a temperature range from -70°F to +900°F; the epoxy resin mainly improved the strength over the lower half of the temperature range. Shear strength at 900°F was about 100 psi.

Manuscript released by author August 1957 for publication as a WADC Technical Report.

June 1956

SUBJECT: DEVELOPMENT OF NON-DESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS  
INVESTIGATOR: J. S. Arnold  
CONTRACT: AF 33(616)-2035  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: An ultrasonic technique for the evaluation of structural adhesive bonds (The STUB-meter) is being developed. The behavior of a ferroelectric transducer, when mechanically coupled to a test specimen, is affected by the structural properties of the test specimen. The design and construction of a portable laboratory model of the STUB-meter are described, and a plausibility argument for the existence of a relationship between bond strength and the STUB-meter behavior is presented. An evaluation program that will be carried out in cooperation with organizations in the aircraft industry is described.

WADC TR 54-231 Pt 5  
ASTIA Document No. AD 118083  
OTS Release

February 1957

SUBJECT: DEVELOPMENT OF NON-DESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS  
INVESTIGATOR: J. S. Arnold  
CONTRACT: AF 33(616)-2035  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: An ultrasonic technique for the evaluation of structural adhesive bonds (the STUB-meter) is being developed and tested. The operation of the instrument is based on the empirically observed fact that the behavior of a ferroelectric transducer, when mechanically coupled to a test specimen, is affected by the structural properties of the test specimen. To define more closely the scope of the technique, an evaluation program is being carried out in cooperation with organizations in the aircraft industry. Initial data have already provided improved means of choosing optimum frequency ranges. Laboratory development of the STUB-meter has included work on probes for curved surfaces and for standard lap shear specimens; electrodes with improved wear resistance; visualization of vibration modes; effects of loading; and improved circuitry.

WADC TR 55-271 Pt II  
ASTIA Document No. AD 110588  
OTS Release

November 1956

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT INORGANIC POLYMER STRUCTURAL ADHESIVES

INVESTIGATOR: Harold H. Levine  
CONTRACT: AF 33(616)-2555  
CONTRACTOR: Quantum, Incorporated  
ABSTRACT: This report describes the progress achieved towards the development of an elevated temperature resistant inorganic polymer structural adhesive.

Increasingly severe thermal operating conditions have been engendered by high aircraft speeds. It is necessary to develop a structural adhesive that can satisfactorily withstand temperatures up to 1000°F.

An inorganic adhesive stable at 800°F, with a room temperature shear strength of 285 lb./sq. in. has been obtained from the ammeline-phosphorus pentoxide reaction product.

The use of titanium dioxide as a primer and dimethylformamide as an extractive solvent enabled the above shear strength to be obtained from an original value of 65 lb./sq. in.

Evidence was obtained to indicate that the adhesion was a result of chemical interaction between the adhesive and the stainless steel surface.

The success of the research to date indicates the necessity of further work to increase the adhesive and flexural strength by chemical methods. This should be done without sacrificing the present heat resistance and preferably with increasing the present thermostability.

WADC TR 55-491  
ASTIA Document No. AD 97316  
OTS Release

September 1956

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES  
INVESTIGATOR: Richard M. Spriggs  
Henry G. Lefort  
Dwight G. Bennett  
CONTRACT: AF 33(616)-2556  
CONTRACTOR: University of Illinois  
ABSTRACT: Twenty-three ceramic adhesives of the ceramic-oxide, glassy-bond type, including six commercially available enamel frit compositions, were evaluated during the contract period. Evaluation was based, primarily, on the results of room and elevated temperature shear strength tests. Best results were obtained with ceramic adhesive UI 117-50. When used with 28 mesh stainless steel screen as a carrier, this adhesive gave shear strengths of up to 3680 psi when tested at 800°F. Twelve ceramic adhesives of the cermet type were tested in shear at room and elevated temperatures. Of these, only the adhesive which contained 30% metallic cobalt incorporated in a lead borosilicate glass matrix appeared to offer promise. Thirteen ceramic adhesives of the

air-setting, temperature-resistant type were also evaluated. Adhesive A-1 was the only air-setting adhesive which approached the target property of 1000 psi at 1000°F, giving shear strengths of up to 1400 psi at 1000°F after a selected curing treatment to 600°F. Its resistance to moisture, however, was quite low. The oxychloride and oxysulfide adhesives investigated developed nominal shear strengths of 200 to 250 psi at room temperature and maintained these strengths to an upper temperature limit of 600°F.

WADC TR 55-491 Pt II  
ASTIA Document No. AD 110736  
OTS Release

January 1957

**SUBJECT:** RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES  
**INVESTIGATOR:** Henry G. Lefort  
Richard M. Spriggs  
Dwight G. Bennett  
**CONTRACT:** AF 33(616)-2556  
**CONTRACTOR:** University of Illinois  
**ABSTRACT:** The study of ceramic compositions as adhesives was activated due to the need for materials to act as adhesives for metals at elevated temperatures and to possess high shear strengths for extended periods of time at such elevated temperatures.

The object of the investigation was to develop high temperature resistant structural adhesives for type 301 and 302 stainless steel or other desired alloy metals, particularly 17-7 PH, from ceramic-oxide, glassy-bonded coatings, cermets with sintered metal bonds, air-setting, temperature-resistant silicates, aluminates, oxychlorides, oxysulfides, and ceramic-oxide, resin-bonded coatings.

Twenty-four ceramic adhesives of the ceramic-oxide, glassy bond type, including three commercially available porcelain enamel ceramic adhesives, were evaluated for use at room and elevated temperatures. Evaluation was based on shear strength tests using type 302 stainless steel. Several of these ceramic adhesives were used to bond small type 17-7 PH stainless steel honeycomb sandwiches. Type 17-7 PH stainless steel was evaluated for shear strength using ceramic adhesive UI 117-50. Various methods of specimen and ceramic adhesive preparation and fabrication were investigated for increase in shear strength including knurled overlap areas, mechanical scouring of overlap areas, chemical pickling and nickel plating of overlap areas, and the use of various foils and screens as carriers. Stainless steel screen of 325 mesh when used as a carrier gave an average shear strength of 1160 psi at 800°F. Copper screen of 60 mesh gave an average shear strength of 1180 psi at 800°F when used as a carrier. Porcelain enamel ceramic adhesives of thermal expansions approaching that of ingot iron shear specimens generally gave the highest shear strengths. Resorcinol-formaldehyde-Portland cement adhesives did not show great promise



as adhesives, especially at elevated temperatures. Stress rupture determinations using ceramic adhesive UI 117-50 on type 302 stainless steel shear strength specimens yielded results of 700 psi for 171 hours with no failure, and 800 psi for 22 hours with failure at room temperature. At 600°F, the adhesive withstood 600 psi for 500 hours without failure.

WADC TR 56-239

September 1956

ASTIA Document No. AD 97292

OTS Release

SUBJECT: TENSILE STRENGTH OF ADHESIVE BONDS IN SANDWICH WITH ALUMINUM FACINGS AND ALUMINUM HONEYCOMB CORES

INVESTIGATOR: V. C. Setterholm  
H. W. Eickner  
E. W. Kuenzi

CONTRACT: AF 33(616)-56-9

CONTRACTOR: Forest Products Laboratory

ABSTRACT: Results of tensile tests of eight adhesive systems for bonding aluminum alloy facings to aluminum honeycomb cores of various cell sizes and foil thicknesses are presented. Analysis of these results shows that the tensile strength of bonds to honeycomb cores with any one adhesive system was found to be mainly dependent on the available fillet length.

Seven of the adhesive systems were adequate for sandwich with cores of 0.001-inch 3003H19 aluminum alloy foil, and 2 adhesives were adequate for sandwich with cores of 0.002-inch foil. None of the adhesives were adequate for producing maximum facing stresses in sandwich with cores of foil thicker than 0.002 inch. Five of the adhesives were strong enough to cause 0.001-inch foil to fail in tension, but none were strong enough to cause failure in thicker foils. Good bonds were not obtainable with one tape adhesive used on cores with cutting burrs on the cell wall ends unless an adequate liquid primer was applied to the core.

WADC TR 56-320

April 1957

ASTIA Document No. AD 118228

SUBJECT: RESEARCH ON STRUCTURAL ADHESIVE PROPERTIES OVER A WIDE TEMPERATURE RANGE

INVESTIGATOR: H. R. Merriman

CONTRACT: AF 33(616)-2620

CONTRACTOR: The Glenn L. Martin Company

ABSTRACT: The widespread increased use of adhesive bonded construction in military aircraft requires that the mechanical properties of adhesives be determined over a wide temperature range in order to determine the useful operating temperature range.

The mechanical properties of adhesive bonded aluminum joints were determined using standard test procedures over a range of temperatures from -100°F to +800°F. The effect of exposure at test temperature for various periods of time was also determined. The effect on the properties of using a different adherend was determined by duplication of some of the test conditions on stainless steel lap joint specimens. Nine general purpose adhesives, AF-6, PA-101, Plastilock 608, Metlbond 4021, FM-47 Liquid, FM-47 Film, Redux E (Type R), Cycleweld 55-20 and Epon VIII; and two high temperature adhesives, Shell 422 and HT-20, were tested.

The program was confined to the testing of standard specimens, bonded in accordance with exact procedure specified at the beginning of the contract by the adhesive manufacturers to obtain the optimum properties over a wide temperature range. Tensile Shear, creep rupture, tensile, impact, bend, cleavage, fatigue and peel tests were conducted.

Lap specimens of general purpose adhesive decreased in strength after 1/2 hour exposure at test temperature compared to test values obtained immediately after reaching test temperatures. Further exposure in general produced increased strength over the initial values obtained for the bonded joints.

WADC TR 56-410  
ASTIA Document No. AD 118203  
OTS Release

April 1957

SUBJECT: RESEARCH ON ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS  
INVESTIGATOR: Roy H. Moulton  
William E. St. Clair  
CONTRACT: AF 33(616)-2862  
CONTRACTOR: Koppers Company Inc.  
ABSTRACT: A study has been made on the formulation of room temperature, low-pressure cured metal-to-metal adhesives, based on the use of glycidyl ethers of resorcinol resins. Various formulations have been found which meet all of the primary strength requirements for tensile shear strength at various temperatures, bend strength, creep-rupture strength and fatigue resistance.

Resistance to salt spray and soaking in water has been tested at Wright Air Development Center on one formulation and found to be unsatisfactory for the former but excellent with the latter.

The preferred formulations resulting from this study have been those with pot lives of one to three hours as obtained by use of solid diamines as curing agents. Longer pot lives of four hours or more have been obtained, but only with the sacrifice of physical strength characteristics.

February 1957

SUBJECT: EFFECTS OF NUCLEAR RADIATION ON STRUCTURAL ADHESIVE BONDS

INVESTIGATOR: Robert S. Arlook, 1/Lt  
Douglas G. Harvey, 1/Lt

ABSTRACT: Nine commercially available metal-to-metal adhesives were investigated to determine the effect of gamma radiation upon their strength properties. Standard 2024-T3 alclad aluminum alloy lap joint specimens were prepared, and three series of exposures were made at the MTR gamma facility. The approximate dosages were  $9 \times 10^7$ ,  $3 \times 10^8$ , and  $9 \times 10^8$  roentgens. Tensile shear and bend tests were conducted at room temperature, at 180°F, and at 260°F. Two adhesives were also tested at 500°F. Fatigue tests were performed at room temperature.

Only one adhesive was essentially unaffected by gamma radiation at all testing temperatures. The remaining adhesives were affected to varying degrees.

WADC TR 56-533  
ASTIA Document No. 118153  
OTS Release

March 1957

SUBJECT: ELEVATED TEMPERATURE RESISTANT SILICONE STRUCTURAL ADHESIVES  
FOR METALS

INVESTIGATOR: Frank J. Riel, Jr.  
M. Bruce Smith

CONTRACT: AF 33(616)-3007

CONTRACTOR: Narmco, Incorporated

ABSTRACT: During the period covered by this report, two possible methods of approach to the synthesis of a new silicon containing polymer suitable for a high temperature adhesive were investigated.

The first type of materials investigated, polymers made from polyhydroxy compounds and halosilanes, proved to be too susceptible to hydrolysis to be useful as adhesives.

The second line of approach involved the synthesis of polysiloxane and polysilane resins containing tolyl groups, followed by oxidation of the tolyl groups to polar carboxyphenyl groups. A number of such resins were prepared, identified, and evaluated.

Evaluation of the carboxyphenyl polysiloxanes showed them to be better high temperature resistant laminating resins than high temperature resistant metal-to-metal adhesives when compared with conventional commercially available silicones. Ultimate flexural strengths of fiber glass reinforced laminates obtained from the system as a whole, were 36,300 psi tested at room

temperature and 16,700 psi tested at 500°F. Ultimate tensile shear values of aluminum-to-aluminum bonds, also obtained from the system as a whole, were 1,322 psi tested at room temperature and 632 psi tested at 500°F.

Heat degradation, of laminate flexural strengths and metal-to-metal bond strengths, was especially evident in tests at R. T. after exposure to 500°F, but not so evident in tests at 500°F after exposure to 500°F.

Little effort was expended in the study of carboxyphenyl polysilanes.

WADC TR 56-650  
ASTIA Document No. AD 118193  
OTS Release

April 1957

SUBJECT: DEVELOPMENT OF METAL-BONDING ADHESIVE WITH IMPROVED HEAT RESISTANCE  
INVESTIGATOR: J. M. Black  
R. F. Blomquist  
CONTRACT: DO 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Studies were conducted on an experimental phenol-epoxy resin tape adhesive, FPL-878, to determine the effects of glass and asbestos supporting mediums and of inorganic fillers on the resistance of bonded aluminum joints to aging at 500°F.

Aluminum-to-aluminum bonds with a tape adhesive employing a supporting medium of either glass cloth No. 112 or No. 120 and with either a heat cleaned or Volan A finish were superior in resistance to aging at elevated temperatures to all other fabrics evaluated. The most promising of the asbestos materials was a felt product, Pyrotex No. 9526-RBI. All lap-joint bonds made with tape adhesive formulations, however, were lower in resistance to thermal aging than bonds made from the liquid adhesive.

Tests on antimony trioxide used as an inorganic filler in tapes of both FPL-878 adhesive and a straight epoxy resin adhesive showed this material was the best of the filler materials tested for resisting thermal degradation and also contributed to increased joint strength.

#### ANALYSIS AND MEASUREMENTS

WADC TR 55-255

April 1956

SUBJECT: PRODUCTION OF METALLIZED FIBERS FOR DIPOLE CHAFF  
INVESTIGATOR: C. L. Emerson, Jr.  
George E. Niles

WADC TR 53-373 Sup 4

CONTRACT: AF 33(616)-2604  
CONTRACTOR: Emerson & Cuming, Inc.  
ABSTRACT: The development of a metallized nylon fiber suitable for dipole chaff has been completed. The finished fiber is approximately 2.9 mils in diameter. It consists of a nylon monofilament 2.8 mils in diameter coated first with silver and then with nylon deposited from solvent. The electrical resistance is continuously less than 15 ohms/inch. Abrasion resistance and non-clumping is good. The cost of the packaged product is less than 0.01¢/foot of length.

WADC TN 55-302  
OTS Release

April 1957

SUBJECT: ACCESSORY EQUIPMENT AND PROCEDURES FOR USE OF A 1500 CURIE COBALT-60 GAMMA-RAY SOURCE  
INVESTIGATOR: Marvin C. Atkins, 1/Lt  
ABSTRACT: The Materials Laboratory is using a 1500 curie cobalt-60 source to investigate the effects of gamma radiation on materials. The source is a cylinder, 1.78 in. I.D. x 2.31 in. O. D. x 13.5 in. long. It is shielded by a lead container which is mounted on a table behind a concrete block wall. A rotating-beam hoist is used to change samples. Other items of accessory equipment include specialized plugs for the container, additional sample canisters, and a cooling system.

Measurements have been made of scattered radiation from the source. A standard operating procedure for use of the source has been established. This procedure has significantly reduced the probability of personnel exposure to harmful radiation.

WADC TR 55-495 Pt I  
ASTIA Document No. AD 110404

September 1956

SUBJECT: MEASUREMENTS OF THERMAL PROPERTIES  
INVESTIGATOR: I. B. Fieldhouse A. N. Takata  
J. C. Hedge T. E. Waterman  
J. I. Lang  
CONTRACT: AF 33(616)-2903  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The objective of this program was the measurement of the high temperature thermal properties of materials. The materials investigated were electrolytic copper, grade A nickel, arc melted unalloyed molybdenum, graphite type 3474D (Speer Carbon Company), graphite type GBE (National Carbon Company), and sintered tantalum. The thermal conductivity, specific heat, and linear coefficient of thermal expansion were measured from 1000°F to 3000°F or the melting point of the material, whichever was lower. All measurements were made in the Heat Transfer laboratory of the Propulsion and Fluid Mechanics Research

WADC TR 53-373 Sup 4

Department, Armour Research Foundation of Illinois Institute of Technology.  
Both the experimental measurements and the results of the conversion of these  
measurements to the desired physical properties are given.

WADC TR 55-495 Pt II  
ASTIA Document No. AD 110510

November 1956

SUBJECT: MEASUREMENTS OF THERMAL PROPERTIES  
INVESTIGATOR: I. B. Fieldhouse J. I. Lang  
J. C. Hedge T. E. Waterman  
CONTRACT: AF 33(616)-2903  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The objective of Part II of this program was the measurement  
of the thermal conductivity and specific heat of liquid electrolytic copper up  
to 3000°F. All measurements were made in the Heat Transfer laboratory of the  
Propulsion and Fluid Mechanics Research Department, Armour Research Foundation  
of Illinois Institute of Technology.

WADC TR 55-495 Pt III  
ASTIA Document No. AD 110526

November 1956

SUBJECT: MEASUREMENTS OF THERMAL PROPERTIES  
INVESTIGATOR: I. B. Fieldhouse  
J. C. Hedge  
T. E. Waterman  
CONTRACT: AF 33(616)-2903  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The objective of Part III of this program was the measurement  
of the thermal conductivity of sintered tantalum (Fansteel Metallurgical  
Corporation), graphite type 7087 (Speer Carbon Company) and graphite type GBH  
(National Carbon Company) from 1000°F to 3000°F. All measurements were made  
in the Heat Transfer Laboratory of the Propulsion and Fluid Mechanics Research  
Department, Armour Research Foundation of Illinois Institute of Technology.

WADC TR 55-496  
ASTIA Document No. AD 97185

August 1956

SUBJECT: THERMAL CONDUCTIVITIES, HEAT CAPACITIES, AND LINEAR THERMAL  
EXPANSION OF FIVE MATERIALS  
INVESTIGATOR: C. F. Lucks  
H. W. Deem  
CONTRACT: AF 33(616)-2902  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Thermal-conductivity measurements on copper, chromium, molyb-  
denum, Grade GBH graphite, and Grade 7087 graphite are reported for the tempera-  
ture range of about 200 to 1700°F. Calibration of equipment to extend the  
measurements to 3000°F. is presented.

WADC TR 53-373 Sup 4

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Heat-capacity measurements have been made on chromium, molybdenum, Grade GBH graphite, and Grade 7087 graphite from 32 to 3000°F., and on copper from 32 to 1950°F.

Measurements on the linear thermal expansions of chromium, molybdenum, Grade GBH graphite, and Grade 7087 graphite from 68 to 3000°F., and of copper from 68 to 1800°F. are reported.

WADC TR 55-496 Pt II  
ASTIA Document No. AD 118168

March 1957

SUBJECT: THERMAL CONDUCTIVITIES, HEAT CAPACITIES, AND LINEAR THERMAL EXPANSION OF FIVE MATERIALS Part II. Density and Thermal Conductivity of Molten Copper

INVESTIGATOR: C. F. Lucks  
H. W. Deem

CONTRACT: AF 33(616)-2902

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: The density of molten electrolytic tough-pitch copper was determined for the temperature range 2000 to 3000°F. The thermal conductivity of molten electrolytic tough-pitch copper was measured at 2161 and 2329°F.

WADC TN 56-196

May 1956

SUBJECT: GAMMA DOSIMETRY PROPERTIES OF LINEAR POLYETHYLENE BY INFRARED ANALYSIS

INVESTIGATOR: James H. Tillotson, 1/Lt  
Nora E. Srp

ABSTRACT: Changes in the molecular structure of long chain polymers by infrared analysis gives an indication of damage to material after its exposure to nuclear radiations. The intensity changes of the 5.8 and 11.0 micron infrared absorption bands in gamma irradiated Marlex 50, a high density linear polyethylene, are analyzed and presented as a measure of relative gamma dosimetry. All radiation exposures were conducted at the National Reactor Test Station, Idaho Falls, Idaho. The infrared absorption spectra were recorded at the Wright Air Development Center using a Baird double beam recording spectrophotometer. The graphs of optical density versus gamma dosage are believed to represent in a realistic manner the gamma dosimetry properties of the polyethylene used in this investigation.

WADC TR 56-222 Pt I  
ASTIA Document No. AD 110458  
OTS Release

October 1956

SUBJECT: DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS Part I. Techniques for Measurement of Total Normal Emissivity and Reflectivity with Some Data on Copper and Nickel

WADC TR 53-373 Sup 4

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INVESTIGATOR: Howard T. Betz  
O. H. Olson

B. D. Schurin  
James C. Morris

CONTRACT: AF 33(616)-3002

CONTRACTOR: Armour Research Foundation

ABSTRACT: Equipment has been designed, constructed, and calibrated for the measurement of total normal emissivity in the range  $-300^{\circ}\text{F}$  to  $+3000^{\circ}\text{F}$ . The procedure consists in comparing the total normal radiance of a sample to that of a comparison blackbody, the ratio of the signals being taken as the emissivity. A thermistor detector is used in conjunction with a folded optical system to record the radiation which is chopped at 16 cps. Measurements of the total normal emissivity for a limited number of samples have been completed. The normal spectral emissivity measurements are obtained by an optical pyrometer method, which relates the true temperature of a sample to its apparent brightness temperature. A Leeds and Northrup disappearing filament pyrometer is used to make these measurements.

An integrating sphere reflectometer is used to measure spectral reflectivity from 0.3 microns to 3.0 microns. This utilizes a Perkin-Elmer monochromator, a comparison type integrating sphere and a PbS cell detector. Spectral reflectivity data can be combined with solar spectral energy data for any location to obtain the solar absorptivity at that point.

WADC TR 56-263

September 1956

ASTIA Document No. AD 97280

OTS Release

SUBJECT: POLAROGRAPHIC DETERMINATION OF GALLIUM IN ALUMINUM AND  
ALUMINUM ALLOYS

INVESTIGATOR: George W. Latimer, Jr., 2/Lt  
Charles D. Houston

ABSTRACT: Polarographic determinations of gallium have been limited to concentrations less than  $3 \times 10^{-5}$  molar because of the interference of the hydrogen wave. By adapting a method utilized by Williard and Dean to determine aluminum, the interference caused by the evolution of hydrogen is eliminated and gallium concentrations up to  $15 \times 10^{-5}$  molar can easily be determined. The gallium is separated from aluminum and most interfering ions by ethyl ether extraction from 5M HCl. The ether is evaporated and the gallium is complexed with Eriochrome Violet. This complex has a polarographic wave beginning at  $-0.3\text{v}$  which is directly proportional to the concentration of gallium.

WADC TR 56-297

October 1957

ASTIA Document No. AD 110435

SUBJECT: A MODIFIED ICE CALORIMETER

WADC TR 53-373 Sup 4

12



INVESTIGATOR: Myron W. Belaga, 1/Lt  
Robert F. Trapp, 1/Lt

ABSTRACT: The design, construction, and calibration of a Bunsen type ice calorimeter and accompanying furnace have been completed.

The calorimeter was electrically calibrated and had a calibration constant of 64.56 calories per gram of mercury change relative to an accepted value of 64.631 calories per gram of mercury as determined by the National Bureau of Standards.

The enthalpy of standard aluminum oxide (supplied by NBS) was determined over a temperature range of 100° - 1650°F and the results were compared with those of the National Bureau of Standards. The enthalpy of aluminum oxide was determined within 6% of the standard values.

WADC TR 56-299  
ASTIA Document No. AD 97281  
OTS Release

September 1956

SUBJECT: SIMULTANEOUS POLAROGRAPHIC DETERMINATION OF CADMIUM AND TIN

INVESTIGATOR: George W. Latimer, Jr., 2/Lt  
C. D. Houston  
Keith E. Eubank

ABSTRACT: The development of new and improved platings for copper sheets has led to a need for improved methods of analyzing the plated material. The simultaneous polarographic determination of cadmium and tin provides a rapid method of analyzing these coatings. Analysis of synthetic standards by this method shows that there are very few interfering substances and that this procedure is applicable to a wide range of concentrations with reasonable accuracy.

WADC TR 56-400 Pt I  
ASTIA Document No. AD 118144

March 1957

SUBJECT: THERMAL PROPERTIES OF MATERIALS Part I. Properties of Graphite, Molybdenum and Tantalum to Their Destruction Temperatures

INVESTIGATOR: N. S. Rasor  
J. D. McClelland

CONTRACT: AF 33(616)-2909

CONTRACTOR: North American Aviation, Inc.

ABSTRACT: Thermal expansion, specific heat, and thermal conductivity have been determined from 2000°F to near the destruction temperature of molybdenum (M. P. 4700°F), tantalum (M. P. 5400°F), and four types of graphite (sublimes at 6600°F). To obtain and measure the temperatures required, a carbon tube furnace, a carbon helix furnace, and a photoelectric pyrometer were

were developed. The details of their construction and use are described. The thermal expansion data for the materials investigated show no grossly unusual features. The specific heat of the metals, particularly molybdenum, appreciably exceeded the Dulong-Petit value. However, their thermal conductivities closely followed the Lorenz (Wiedemann-Franz) relation for free electrons. At intermediate temperatures (2000 to 5000 F) the thermal conductivity and specific heat of graphite show no unusual features. However, at higher temperatures large and abrupt increases in specific heat and thermal resistivity occur which are consistent with the occurrence of thermally produced vacant lattice sites. A vacancy concentration of about 0.4 atomic per cent at the sublimation temperature, and a formation energy in the vicinity of 7 ev are indicated.

WADC TR 56-400 Pt II  
ASTIA Document No. AD 118243

April 1957

SUBJECT: THERMAL PROPERTIES OF MATERIALS Part II. Thermal Conductivity of Liquid Copper  
INVESTIGATOR: J. D. McClelland R. C. Dahleen  
N. S. Rasor E. H. Zehms  
CONTRACT: AF 33(616)-2909  
CONTRACTOR: North American Aviation, Inc.  
ABSTRACT: Thermal conductivity of liquid copper from approximately 2550°F to 4050°F is found to be nearly constant at about 68 Btu/ft-hr-° F in a study making use of apparatus and techniques developed for and reported in WADC TR 56-400, Part I.

WADC TR 56-433  
ASTIA Document No. AD 110733  
OTS Release

January 1957

SUBJECT: NEUTRON INDUCED RADIOACTIVITIES IN IF-6 IN-FLIGHT FOOD PACKETS  
INVESTIGATOR: Robert P. Epple  
CONTRACT: AF 33(616)-2616  
CONTRACTOR: Tracerlab, Incorporated  
ABSTRACT: A study has been made of the radioactivities induced by neutron irradiation of IF-6 In-Flight food packets. The results show that an integrated exposure of  $4 \times 10^{14}$  nvt was produced.

Na-24 and K-42 were produced in amounts which are nearly 1000 times the accepted tolerances for these isotopes in the human body.

WADC TR 56-434  
ASTIA Document No. AD 118046  
OTS Release

February 1957

SUBJECT: INFRARED SPECTRA OF CYCLOPENTAMETHYLENEDIALKYL SILANES IN  
THE 2-35 MICRON REGION  
INVESTIGATOR: Gordon D. Oshesky  
Freeman F. Bentley

ABSTRACT: The difficulty in establishing the presence of a ring in organic molecules by chemical means has promoted interest in identifying cyclic structures by their infrared spectra. Saturated cyclic systems have characteristic absorption frequencies arising from the bond stretching and valence angle modes which occur in the fundamental region and the out-of-plane and torsional bending modes which occur below 20 microns. In this study the infrared spectra of a series of cyclic silanes has been correlated with their molecular structure and empirical assignments made for the absorption frequencies characteristic of the heterocyclic ring.

The infrared absorption spectra of 21 cyclopentamethylenedi-alkylsilanes has been obtained in the liquid state over the range 2-35 microns using NaCl and CsBr prisms. The series begins with cyclopentamethylenedisilane and ends with cyclopentamethylenediocetyldecylsilane. Absorption bands at 10.93 to 11.00 microns and 20.20 to 20.85 microns in the spectra of these compounds are tentatively assigned to vibrations arising from the heterocyclic ring.

Absorption bands attributed to the alkyl-silicon linkage and the spectra of these molecules in the long wavelength region are also discussed. All the observed absorption bands are given in a table showing their position and relative intensities.

WADC TR 56-440  
ASTIA Document No. AD 118244

April 1957

SUBJECT: INVESTIGATION OF ISOTOPES FOR AIRCRAFT RADIOGRAPHY  
INVESTIGATOR: Eric T. Clarke  
CONTRACT: AF 33(616)-3042  
CONTRACTOR: Technical Operations, Incorporated

ABSTRACT: To determine if isotope radiography will permit inspection of aircraft components, without the need for disassembly, a study was made of the properties of the isotopes Eu-155, Ce-144, Am-241, Ir-192, and Tm-170 in the radiography of light metal structures. On the basis of ease of production, available intensity, and ability to produce sufficient radiographic contrast in typical aircraft materials, the isotopes Ir-192 and Tm-170 were selected and their characteristics in stainless steel, titanium, aluminum alloy, and magnesium were measured. Radiographic exposure charts and sensitivity data were established for these materials in conjunction with suitable photographic materials, including lead and fluorescent screens. Data are given on production of these isotopes in nuclear reactors.

Results of radiography of actual aircraft are shown which indicate that internal structural detail can be readily made visible, and some types of flaws can be detected, without the need for excessive exposure times. Reproductions of actual radiographs permit comparisons with X-rays.

WADC TR 56-452  
ASTIA Document No. AD 110542  
OTS Release

December 1956

SUBJECT: DESIGN OF A HIGH-INTENSITY GAMMA IRRADIATION FACILITY  
INVESTIGATOR: Marvin C. Atkins, 1/Lt  
ABSTRACT: Construction plans have been prepared for a gamma irradiation facility capable of handling 100,000 curies of cobalt-60. In a facility built according to these plans, the radioactive source would be stored in a water well located in a 12 x 12 foot irradiation room. The source would rest on a platform in the well and be lifted up into the irradiation room for all experiments. The facility would be built underground with almost all shielding being provided by the natural earth. Normal laboratory utilities are provided in the plans, with valves and switches located on a control panel in an adjacent building. A closed circuit television system would be installed for viewing of experiments with the source in the operating position. Experimental apparatus would be equipped with suitable instruments so that general purpose manipulators would not be required. Six construction drawings are included in this report.

WADC TR 56-500  
ASTIA Document No. AD 118273  
OTS Release

May 1957

SUBJECT: SPECTROGRAPHIC ANALYSES OF SOLID TITANIUM  
INVESTIGATOR: Jack A. Winstead, 1/Lt  
ABSTRACT: The purpose of this work was to establish satisfactory excitation conditions for spectrographic analysis of solid titanium and to obtain data from analysis by the established technique.

This work was done in cooperation with the Task Force on Spectrographic Analysis by Direct Metal Excitation. Watertown Arsenal supplied each member of the Task Force seven samples (two specimens each from different locations).

Each specimen was cut in half and then nine analyses were made on each individual piece, totalling thirty-six analyses for each alloy sample. These analyses were made and data included in this report.

January 1957

SUBJECT: ANALYSIS OF THE THERMAL PROPERTIES OF PLASTIC LAMINATES, CORES, AND SANDWICH PANELS  
INVESTIGATOR: Robert F. Trapp, 1/Lt  
ABSTRACT: The thermal properties of plastic laminates, cores, and sandwich panels are presented by grouping data from WADC Technical Reports 54-306 Parts I and II into sets, arranged according to the types of resin used in their construction. Curves are presented which are representative of the resin types for each thermal property. The scatter of data about these mean curves varies from  $\pm 10\%$  to  $\pm 40\%$ .

WADC TR 56-657  
ASTIA Document No. AD 118272  
OTS Release

May 1957

SUBJECT: THEORETICAL ANALYSIS OF THE RESPONSE OF A PROTON RECOIL TYPE NEUTRON DETECTOR  
INVESTIGATOR: Sven R. Hartmann, 1/Lt  
ABSTRACT: A calculation is made to determine the number of recoil protons produced per unit area, by a neutron flux of arbitrary energy, in an infinite hydrogenous slab which reaches but does not pass through an infinite detector slab located immediately behind a hydrogenous slab. A general case is considered for an isotropic neutron flux incident upon the above system in which an arbitrary thickness of some proton absorbing material separates the hydrogenous and detector slabs. A discussion of the results is given indicating how one can control the energy and the energy interval over which the system is sensitive to neutrons. A final calculation is made to determine the response of the system to a collimated neutron flux for an arbitrary orientation of the hydrogenous-detector system. This is done so that the effect of a nonisotropic flux can be determined.

WADC TR 57-38  
ASTIA Document No. AD 118225  
OTS Release

April 1957

SUBJECT: HEAT CAPACITIES OF SYNTHETIC ENGINE OILS AND LUBRICANTS  
INVESTIGATOR: Hyman Marcus  
ABSTRACT: A modified adiabatic type calorimeter, previously designed, built and calibrated (Rev. Sci. Instr 27 p 948, 1956), was used in the measurement of the heat capacities. The variation of the heat capacity as a function of temperature was determined for eleven synthetic fluids conforming to Military Specification MIL-L-7808 and the data are presented graphically.

The expressions for the heat capacity were derived from a least squares statistical analysis of the time-temperature data and an attempt was made to develop a heat capacity envelope of all the data obtained and calculate a median curve. No comparison or analysis was made of each set of individual data nor were any set of data identified with individual products measured.

### BIOCHEMISTRY

WADC TR 56-208  
ASTIA Document No. AD 97183  
OTS Release

August 1956

SUBJECT: THE EFFECTS OF FUNGUS GROWTH AND MOISTURE UPON THE STRENGTH PROPERTIES OF REINFORCED PLASTICS

INVESTIGATOR: R. C. Tomashot  
E. L. Hamilton

ABSTRACT: The effects of fungus growth and moisture upon the strength properties of several reinforced plastic laminates were investigated. Material variations included the type of reinforcement, the chemical type of resin, and the finish on the glass fabric reinforcement. Of the material variations, the type of reinforcement and finish on the glass fabric were significant in regard to both the amount of fungus growth and the reduction of the strength properties. The effect of fungus growth upon the strength properties was not considered significant as compared to the effect caused by the presence of moisture.

WADC TR 56-252  
ASTIA Document No. AD 110711  
OTS Release

January 1957

SUBJECT: WEATHERING RESISTANCE OF FUNGICIDAL VINYL COATED COTTON FABRICS

INVESTIGATOR: John C. Saylor, Jr. 1/Lt.

ABSTRACT: The fungicide, dehydroabietyl ammonium pentachlorophenoxide, formulated in experimental yellow and olive drab vinyl coatings on cotton fabric has been evaluated for resistance to weathering. A comparison has been made with copper 8-hydroxyquinoline formulated in an experimental olive drab vinyl coating and with an olive drab USAF stock vinyl coated fabric containing the same fungicide. The evaluations were based on breaking and tearing strength after outdoor weathering at the Alaska, Florida, New Mexico, and Wright-Patterson AFB exposure sites and on color change after weathering. The amount of

breakdown in strength of the materials evaluated in this study has been correlated with the amount of light energy received from the sun.

The dehydroabietyl ammonium pentachlorophenoxide treated coated fabrics were not acceptable as compared to the coated fabrics with copper 8-hydroxyquinoline which maintained satisfactory strength and color after outdoor exposure. It was observed that shrinkage of the coated fabrics occurred during outdoor weathering.

WADC TR 56-384 Pt I  
ASTIA Document No. AD 118322  
OTS Release

May 1957

SUBJECT: AN EVALUATION OF FUNGICIDAL TREATMENTS IN COTTON CARGO PARACHUTE WEBBINGS STORED AT WRIGHT AIR DEVELOPMENT CENTER  
INVESTIGATOR: Emma F. Little  
ABSTRACT: Five fungicidal formulations, with or without water repellents, were applied to four types of cotton cargo parachute webbings. Three of the formulations contained copper 8-quinolinolate, one contained dihydroxy-dichloro-diphenyl methane, and one contained dehydroabietyl ammonium acetate.

In order to determine the efficiency of these formulations in the webbings, five evaluations were made: (1) soil burial, (2) simulated tropical storage, (3) shelf storage under laboratory conditions, (4) agar plate exposure, and (5) agar plate exposure following laboratory shelf storage. Cellulolytic fungi used as test organisms were Aspergillus terreus, Myrothecium verrucaria, and Chaetomium globosum. Conclusions were based on general appearance and breaking strength data derived from tests of webbings and webbing threads, chemical analyses and visual observations. All formulations of copper 8-quinolinolate evaluated in this research were equally effective in webbing, providing the minimum metallic copper content as copper 8-quinolinolate was 0.14% by weight of the treated material. Further, webbings containing a minimum of 0.9% dihydroxy-dichloro-diphenyl methane by weight of the treated material, were equally fungus resistant. None of the fungicidal treatments caused significant tenderizing of the webbings after one year in simulated tropical or in laboratory shelf storage conditions. Webbings containing dehydroabietyl ammonium acetate were not resistant to fungi in soil and this fungicide caused primary skin irritation to human beings. Therefore, this fungicide was not considered satisfactory for USAF use.

WADC TR 56-431  
ASTIA Document No. AD 97279  
OTS Release

September 1956

SUBJECT: AN ELEMENTARY STATISTICAL APPROACH TO MICROBIOLOGICAL RESEARCH  
INVESTIGATOR: James L. Mahan

**ABSTRACT:** The statistical approach is illustrated by making an analysis of data obtained from an experiment which was designed to evaluate a number of commercial fungicidal treatments on cotton webbing. The treatment and analysis of the data consists of the following:

1. A statistical summary of the data consisting of the Mean, Standard Deviation, and the Standard Error of the Mean.

2. An Analysis of Variance of the data. In addition, the Newman-Keuls test is used to arrange the significant treatment combinations according to their effectiveness.

3. The results of the analysis of variance and the Newman-Keuls test are considered in relation to information concerning the phenomena associated with the use of fungicides.

4. The information obtained from the analysis of variance and the statistical summary of the data are used to estimate the minimum sample size required for tests of fungicides according to specification requirements.

5. The importance of random sampling is discussed briefly and illustrated.

6. The importance and value of the statistically designed experiment are discussed. References to selected books concerning experimental design are given.

WADC TR 56-480 Sup 1  
ASTIA Document No. AD 110441  
OTS Release

October 1956

**SUBJECT:** EVALUATION OF FUNGUS RESISTANCE OF COTTON THREAD TREATED WITH  
SELECTED FUNGICIDAL FORMULATIONS Supplement 1 Statistical  
Analysis of Data

**INVESTIGATOR:** Arthur Rose  
Thomas B. Hoover

**CONTRACT:** AF 33(600)-26749 S/A 3

**CONTRACTOR:** Applied Science Laboratories, Inc.

**ABSTRACT:** Under Supplemental Agreement No. 3 to Contract AF 33(600)-26749 Applied Science Laboratories, Inc., has made a statistical analysis of the data obtained in an evaluation of the fungus resistance of cotton thread treated with selected fungicides. The data and a description of the test program were reported in WADC TR 56-480. The statistical treatment includes analysis of variance of breaking strengths of threads exposed to the fungus tests and also of the corresponding unexposed control threads. The direct effects of each of the main factors (color, finish, fungicidal agent, level of concentration of fungicide, and test method) upon the loss in breaking strength are evaluated and confidence limits are estimated.



## CERAMICS

WADC TR 56-329  
ASTIA Document No. AD 118195  
OTS Release

April 1957

SUBJECT: IMPROVEMENT OF THE IMPACT RESISTANCE OF CERMETS

INVESTIGATOR: A. C. Pezzi

H. P. Kling

CONTRACT: AF 33(616)-2911

CONTRACTOR: Sylvania Electric Products, Inc.

ABSTRACT: This program investigated the possibility of improving the impact resistance of cermets by the application of a well bonded ductile metallic coating to the surface. Commercially available bars of K161B were used as a base cermet. Coating methods investigated included diffusion bonding of electrodeposited nickel, infiltration of porous sintered coatings, direct application of high temperature brazes, diffusion bonding to shaped tubing and vacuum casting. The application of 0.018" of electrodeposited nickel, bonded by a suitable vacuum heat treatment, raised the impact strength from values of 2.56 and 3.36 in-lbs at room temperature and 1800°F respectively to values of 21.48 and 18.96 in-lbs at the same temperatures. Limited test results indicated that diffusion bonding of shaped tubing also raised the impact strength appreciably. The infiltrated coatings showed promising structures when examined metallographically but the impact strength was essentially unchanged with the compositions used. It is possible that other compositions might be more beneficial. The direct application of brazing alloys was deemed unsatisfactory. The vacuum cast coatings had interesting microstructures but were not evaluated.

WADC TR 56-521  
ASTIA Document No. AD 118224  
OTS Release

April 1957

SUBJECT: PREPARATION OF PROTECTIVE COATINGS BY ELECTROPHORETIC METHODS

INVESTIGATOR: Allen C. Werner

Harold N. Barr

James J. Shyne

Bernard Triffleman

CONTRACT: AF 33(616)-3118

CONTRACTOR: Vitro Laboratories

ABSTRACT: An investigation was conducted of the preparation of oxidation-resistant coatings for molybdenum by electrophoretic methods. Multilayer coatings of 80% nickel-20% chromium and nickel-bonded chromium carbide provided good static air-oxidation resistance. Inclusion of a 50% dense nickel-chromium layer resulted in satisfactory ballistic impact resistance. Erosion was fair. All the test panels failed under severe thermal shock.

Electrokinetic impregnation of graphite to provide increased oxidation resistance was unsuccessful, a maximum impregnation of 2.1% by weight being obtained. The effect of varying suspension concentration, impregnation voltage, periodic reversal of the current, activators for the graphite and the suspensions and the use of ultrasonic energy were studied. Impregnation up to 2.6% by weight was obtained by pumping a slurry through graphite without application of any EMF.

WADC TR 57-32  
ASTIA Document No. AD 118278  
OTS Release

May 1957

**SUBJECT:** A STUDY OF HOT PRESSING OF BERYLLIA ROCKET NOZZLES

**INVESTIGATOR:** Chester A. Bielawski  
Theodore M. Harkulich  
Russell E. Long

**CONTRACT:** AF 33(616)-56-23

**CONTRACTOR:** The Brush Beryllium Company

**ABSTRACT:** A preliminary investigation was conducted on the variables of particle size, temperature, pressure, and time as related to the fabrication of beryllia rocket-nozzle throat inserts by hot pressing. Results of this work were applied toward the fabrication of twenty, 100-pound-thrust throat-insert specimens which were submitted for evaluating the feasibility of using this material in uncooled rocket motors. The twenty specimens were composed of four groups representing variations in starting materials with regard to particle size and purity and variations in fabrication procedures, including time, temperature, and heat treatments, which affected the final structure.

WADC TR 57-39  
ASTIA Document No. AD 130754  
OTS Release

May 1957

**SUBJECT:** CERAMIC REINFORCED ALLOYS AND PLATED CERMETS

**INVESTIGATOR:** M. T. Curran R. S. Truesdale  
R. P. Riegert J. R. Tinklepaugh  
R. K. Francis

**CONTRACT:** AF 33(616)-2414

**CONTRACTOR:** The New York State College of Ceramics

**ABSTRACT:** A precision casting technique was developed for the reinforcement of super alloys with ceramic rods. Procedures were found for the electroplating of cermets with nickel and chromium to improve their impact resistance and for the chemical plating of nickel on titanium carbide grains. The effect of molybdenum as an alloying agent in the cermet binder was observed.

SUBJECT: HOT PRESSING OF BERYLLIA ROCKET NOZZLES  
INVESTIGATOR: J. K. Evans  
G. D. Kriebel  
CONTRACT: AF 33(616)-3699  
CONTRACTOR: The Beryllium Corporation  
ABSTRACT: The effects of time, temperature, pressure, and particle size on the density of hot pressed beryllium oxide were investigated. Experimental conditions and densities obtained are tabulated for two types of beryllium oxide and their mixtures. A description of the equipment, procedures, and starting materials is included. The fabrication of rocket nozzles according to the best procedures is described. Photomicrographs of hot pressed beryllia of maximum density are shown. A limited study of the machining of hot pressed beryllia is included.

### COATINGS

WADC TR 54-325 Pt 3  
ASTIA Document No. AD 118184

April 1957

SUBJECT: RESEARCH FOR LOW-APPLICATION-TEMPERATURE ELECTRICALLY CONDUCTING TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS  
INVESTIGATOR: Leonard H. Mauk  
Robert E. Barrett  
Edwin H. Lougher  
Earl R. Olson  
Harvey L. Goering  
CONTRACT: AF 33(616)-2467  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report describes a continuation of the studies described in WADC Technical Report 54-325, Part 2.

Transparent, conducting films on glass were prepared by simultaneous evaporation of indium and tin and subsequent thermal oxidation of the metal films in air at 150 to 200°C. Films with resistance as low as 17 ohms/square and transmittance as high as 88 percent were prepared. Representative specimens had values of 50 to 100 ohms/square and 80 percent transmittance. Greater reproducibility of film properties was attained as a result of the use of improved techniques and equipment and studies of the effect of varying the process parameters.

Large panels (9 x 9 inches) were filmed, using a rotating substrate and a small source located off the axis of rotation. The transmittance and resistance varied only slightly over the surface. The variation can be reduced by relocation of the source.

A study of magnesium fluoride and silicon oxide overcoatings was made in an attempt to increase the transmittance of the conducting films and to confer mechanical protection. Transmittance increases as high as 13 percent were obtained. The hardness of the overcoatings approached but did not exceed that of the conducting films.

WADC TR 54-527 Sup 1  
ASTIA Document No. AD 106911  
OTS Release

June 1956

SUBJECT: THE DEVELOPMENT OF A PROTECTIVE COATING RESISTANT TO NITRIC ACID AND HYDROCARBONS

INVESTIGATOR: D. F. Siddall R. Garling  
E. Hillier M. Gunther

CONTRACT: AF 33(616)-150

CONTRACTOR: The United States Stoneware Company

ABSTRACT: X-200 (Kel-F 800) resin, a new fluorinated fuming nitric acid resistant material was formulated as a lacquer and a filleting putty. Application procedures were developed, and resistance of the lacquer coating, the filleting putty, and the complete system in FNA and JP-4 fuel were determined.

Results indicate that the X-200 system is suitable for application on aircraft metals. When force dried at 300° F, FNA resistance approaches that of fused Kel-F. Resistance of the system when air dried is considerably lower but improves slowly over a long period of time as residual solvent evaporates. The coating system softens in JP-4 fuel after 3 days at 125°F.

WADC TN 55-212 Sup 1  
ASTIA Document No. AD 118081

February 1957

SUBJECT: RESISTANCE OF ORGANIC FINISHES TO ROCKET BLAST

INVESTIGATOR: Alvin M. Savio, S/Sgt.

ABSTRACT: Twenty-five different finishing systems were tested to determine their relative resistance to the corrosive and erosive effects of rocket exhaust gases. The results of these tests indicate that two distinct problem areas are associated with the rocket exhaust effects. These two problems are corrosion and erosion. Standard lacquer and enamel finish systems satisfactorily protect aircraft materials from the corrosive effects of the exhaust gases as long as the coatings are not damaged by abrasion or heat. None of the coating systems tested satisfactorily protect aircraft materials from erosion by rocket exhaust debris and heat.

September 1956

SUBJECT: SERVICE TESTS OF CHEMICAL RAIN REPELLENTS  
INVESTIGATOR: A. M. Savio, S/Sgt.  
D. S. Nantz, 2/Lt.

ABSTRACT: The rain removal systems on many of the current Air Force aircraft are unsatisfactory. Three methods of rain removal are presently being used; jet blast (compressor bleed) method, windshield wipers, and chemical rain repellents respectively. This report is primarily concerned with chemical rain repellents.

Three types of chemical rain repellents were suggested as a low cost, low weight method of providing visibility through rain. These materials were subjected to laboratory and service tests to determine their suitability for current Air Force requirements. The three materials tested were equally satisfactory as rain repellents. However, one material was unsatisfactory for field use due to its toxic and corrosive nature.

Two equivalent materials were recommended for Air Force use, the Snell 1728S developed under Navy contract and Type II developed by the Canadian Research Council, respectively. These materials are easily applied and have suitable durability. They have been procured and are in Air Force stock.

WADC TR 56-591  
ASTIA Document No. AD 118099

March 1957

SUBJECT: ICE ADHESION TESTS ON FILMS OF ORGANIC POLAR MATERIALS  
INVESTIGATOR: W. R. Meyer  
E. F. Foley, Jr.  
CONTRACT: AF 33(616)-3444  
CONTRACTOR: Enthone, Inc.

ABSTRACT: Those compounds which are known to produce superior water displacing films on metals were tested to determine their efficiency in lowering the adhesion of ice to metal surfaces. These tests were made to determine the feasibility of developing ice repellent materials using these compounds or their homologues. The results of ice adhesion measurements on aluminum, steel and cement surfaces coated with these compounds are presented. Most of the compounds tested reduced the force necessary to remove ice from the surfaces tested, but none of them reduced the adhesion below 18 lb/sq. in. It is concluded that none of the materials reduced ice adhesion sufficiently to indicate that practical ice repellent materials could be developed with these compounds as the major active ingredients.

April 1957

STUBJECT: HIGH TEMPERATURE PROTECTIVE COATINGS FOR MAGNESIUM  
INVESTIGATOR: Clyde R. Fitzgibbon  
Edward H. Miller  
Milton A. Glaser  
CONTRACT: AF 33(616)-2890  
CONTRACTOR: Midland Industrial Finishes Company  
ABSTRACT: The objective of the work discussed in this report was to develop an air-drying coating system for magnesium that would have film properties equal to or better than present air-drying finishing systems for this metal -- and which would also withstand temperatures up to 500°F. The major testing requirement for the desired coating system was the ability to maintain film integrity for relatively long periods of time when subjected to a cyclic high humidity-high temperature test.

Three coating systems were developed in this work. They are the "preferred" system and two alternate systems. The alternates, while inferior to the "preferred" system in high humidity-high temperature resistance, are "one-package" materials with better color retention than the "preferred" system. The major drawbacks of the "preferred" system are the lack of color retention at high temperatures and the fact that it would have to be supplied as a multi-package material. The "preferred" system is based on an epoxy-polyamide vehicle. It consists of a calcium chromate pigmented primer, containing 35% pigment, and an aluminumized topcoat. The vehicles on which the alternates are based are:

1. A styrenated acrylonitrile modified alkyd-silicone copolymer resin
2. A mixture of an epoxy ester and a silicone resin.

This work was conducted under USAF Contract No. AF 33(616)-2890, and was performed by the laboratory of the Midland Industrial Finishes Company at Waukegan, Illinois.

#### CORROSION

December 1956

SUBJECT: PERMEABILITY OF BARRIER MATERIALS TO VOLATILE CORROSION  
INHIBITORS AT VARIOUS HUMIDITIES

INVESTIGATOR: J. P. Hohf  
A. A. Mohaupt  
CONTRACT: PO (33-600)-53-4023  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: The comparative permeability of various barrier materials to certain volatile corrosion inhibitors (VCI) was determined at four relative humidities.

Barrier materials containing a metal foil were the most effective for retaining the VCI vapor. Polyester films and materials that conformed to grade C of Specification JAN-B-121 performed well but not as well as the foil barrier materials. Kraft papers proved ineffective for retaining VCI vapors for long time storage, but certain fiberboards performed well enough to be considered for shipping and short periods of storage.

In general, the volatility and transmission rates of the inhibitors increased as humidity increased. An inhibitor consisting of sodium nitrite and urea absorbed the greatest amount of water, while the oily inhibitors absorbed very little water.

WADC TR 55-345  
ASTIA Document No. AD 97141  
OTS Release

July 1956

SUBJECT: DEVELOPMENT OF AN IMPROVED CORROSION INHIBITOR FOR WATER-ALCOHOL SOLUTIONS  
INVESTIGATOR: Dwight B. Conklin  
Brock G. Peacock  
James E. Cole  
CONTRACT: AF 33(616)-2442  
CONTRACTOR: Wyandotte Chemicals Corporation  
ABSTRACT: A corrosion inhibitor was developed for use with alcohol-water injection fluid for aircraft engines. It inhibits corrosion of steel, stainless steel and aluminum alloys, is soluble in methanol, ethanol, water or mixtures of the liquids, and lowers surface tension of the mixtures. Although the inhibitor is chemically compatible with hard water solutions, inhibition efficiency is inversely proportional to water hardness. Solutions made with water of more than 100 parts per million hardness, require an excessive amount of inhibitor for inhibition of corrosion. The inhibitor is a mixture of dicyclohexyl ammonium nitrite, urea and 1-nitropropane in an anhydrous methanol solution. Data gathered from initial screening of 150 corrosion inhibiting compounds is presented in detail.

June 1956

SUBJECT: EVALUATION OF THIN IRON FILMS AS SENSITIVE CORROSION INDICATORS  
INVESTIGATOR: David Roller, A/lc  
ABSTRACT: A requirement exists for a sensitive, easily used, inexpensive, replaceable, and conveniently fabricated direct reading corrosion indicator. Particularly in the field of packaging, a direct reading visual or electrical specimen which could non-destructively determine environmental conditions within all types of containers would be very useful. A preliminary evaluation of the corrosion rates of thin, continuous, adherent vacuum deposited iron films in various humidities to determine their suitability as corrosion indicators is described. Variables investigated included film thickness and film substrate as it affected the corrosion rates in various humidities. It was evident from this work that thin pure iron films corrode readily in high static humidities. Recommendations for further investigations needed to develop a suitable visual or electrical indicator for use in packages are made.

WADC TR 56-310  
ASTIA Document No. AD 110504  
OTS Release

November 1956

SUBJECT: INVESTIGATION OF HYDROFLUORIC ACID AS A CORROSION INHIBITOR  
FOR FUMING NITRIC ACIDS  
INVESTIGATOR: M. J. Keeler  
E. F. Knoll  
CONTRACT: AF 33(616)-2516  
CONTRACTOR: Aerojet-General Corporation  
ABSTRACT: An investigation was made of the effect of small amounts of hydrogen fluoride in inhibiting corrosion by fuming nitric acid. This investigation revealed that corrosion rates of 6061-T6 aluminum and Type 347 stainless steel were generally reduced by a factor varying from about 10 to 100, when an initial HF content of approximately 0.75% by weight was employed. No further reduction in corrosion rate was obtained by increasing the HF content to 2.0%; 0.5% usually provided less inhibition. Therefore 0.75% was selected as the inhibitor concentration for further tests. The effect on the metals of exposure to the inhibited acids was determined in crevices and in stressed specimens, at weld zones, in galvanic couples, in containers with different ullages and with different sizes of vent holes, with various ratios of metal area to acid volume, with acids containing varying amounts of solid matter, and with acids flowing through orifices and impinging against metal at various velocities. The results indicated that acids inhibited with HF are satisfactory for general use. Tests conducted with other metals gave similar results of reduced corrosion in the inhibited acids. Methods for determining HF content and for determining other constituents of acids were also investigated.



February 1957

SUBJECT: A STUDY OF THE VOLUMETRIC PROPERTIES AND THERMAL STABILITY OF  
CONCENTRATED NITRIC ACID

INVESTIGATOR: Webster B. Kay  
Manoj K. D. Sanghvi  
S. Alexander Stern

CONTRACT: AF 33(038)-10381

CONTRACTOR: The Ohio State University Research Foundation

ABSTRACT: 1. The phase relations of the binary and ternary mixtures of nitric acid with nitrogen dioxide and water, containing up to 30% by weight of nitrogen dioxide and 10% by weight of water were determined over the temperature range from 85° to 150°C, and a range of  $V^G/V$  (ratio of the vapor volume to the total volume of the sample) from approximately the bubble point of 0.8. For this purpose, a small sample of a mixture of known composition was maintained under isothermal and isochoric conditions in a glass tube and measurements of the pressure were made at regular intervals of time until physical and chemical equilibria were reached. The reversibility of the decomposition reaction was established and the effects of the  $V^G/V$  ratio, the temperature and the amounts of nitrogen dioxide and water on the pressure were determined within the stated ranges.

2. The pressure, which is primarily due to the oxygen formed as a result of the decomposition, increases with an increase in temperature and with a decrease in the  $V^G/V$  ratio.

3. The pressure is markedly reduced by the addition of nitrogen dioxide and (or) water, particularly at the lower temperatures. On a weight basis, water is more effective in reducing the pressure than nitrogen dioxide.

4. The  $V^G/V$  ratio has a very great influence on the value of the equilibrium pressure of mixtures high in nitric acid content but this influence diminishes with the addition of nitrogen dioxide and water and becomes relatively insignificant in mixtures containing more than 20% of additive (nitrogen dioxide plus water).

5. The effect of temperature on the equilibrium pressure for a given  $V^G/V$  ratio, depends on the proportion of nitrogen dioxide in the additive. In the case of the nitric acid-water mixtures, the equilibrium pressure decreases with increasing water content at all temperatures and all  $V^G/V$  ratios studied, whereas in the case of the nitric acid-nitrogen dioxide mixtures at temperatures above 125°C and for  $V^G/V$  ratios greater than 0.6, the equilibrium pressure is found to increase with the addition of nitrogen dioxide.

6. The rates of attainment of equilibrium are increased by the addition of nitrogen dioxide and decreased by the addition of water. When nitrogen dioxide and water are added in a fixed ratio, the rate constants first show a decrease, pass through a minimum at about 10% of the additive and then increase with a further increase in the additive.

WADC TR 56-414  
ASTIA Document No. AD 118028  
OTS Release

February 1957

SUBJECT: CORROSION AND IGNITION OF TITANIUM ALLOYS IN FUMING NITRIC ACID

INVESTIGATOR: John B. Rittenhouse                      Julia S. Whittick  
                 Nicholas D. Stolica                      David M. Mason  
                 Stephen P. Vango

CONTRACT: AF 33(616)-3066

CONTRACTOR: Jet Propulsion Laboratory

ABSTRACT: The corrosion, ignition reactions, and stress-corrosion cracking of titanium and its alloys resulting from storage in fuming nitric acid (FNA) were studied. The metal samples were stored in the liquid and vapor phases of various concentrations of the system<sup>a</sup>  $\text{HNO}_3\text{-NO}_2\text{-H}_2\text{O}$  for periods of time ranging from 1 hour to 90 days at temperatures ranging from room temperature to 71°C.

The susceptibility to ignition reactions, the tendency toward stress-corrosion cracking, and the corrosion rates of the metal were studied as a function of the chemical composition and temperature of the FNA.

The corrosion-time relationships of two titanium alloys in anhydrous FNA (20%  $\text{NO}_2$ )<sup>b</sup> over a temperature range from 25°C to 71°C were determined. Results of metallographic examinations of the corroded samples to ascertain the corrosion mechanism and the effects of heat treatment of the samples on the corrosion behavior are discussed.

Chemical and X-ray diffraction analyses of the alloys, the FNA used, and the corrosion products developed are reported.

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<sup>a</sup>The symbol  $\text{NO}_2$  designates the equilibrium mixture of  $\text{NO}_2$  and  $\text{N}_2\text{O}_4$ .

<sup>b</sup>Percentages of composition are in weight per cent throughout this report.

DESIGN CRITERIA

WADC TR 52-251 Pt 4  
OTS Release

April 1956

SUBJECT: INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM  
COLUMNS AT ELEVATED TEMPERATURES Part 4. Additional Studies

INVESTIGATOR: R. L. Carlson  
E. G. Bodine  
G. K. Manning

CONTRACT: AF 33(038)-9542

CONTRACTOR: Battelle Memorial Institute

ABSTRACT A summary and an analysis of the results of an experimental study of the creep buckling of columns is presented. The results include investigations of the behavior of short, inelastic columns of the aluminum alloy 2024-T4, of square tubing columns of aluminum alloy 2024-T3, and columns of the stainless steel 17-7PH (THD 1050).

Data from all phases of the investigation indicate that column imperfection variations can cause marked scatter in lifetime. It is concluded that the effect of imperfection variations will exert a strong influence on the evolution of practical methods of designing for creep buckling.

Shanley's time-dependent tangent modulus is applied to the test data, and it is found to be fairly successful in providing an estimate of the possible loss in load capacity with time. It is observed, however, that the agreement with test data can be expected to vary significantly with column imperfection.

The possible existence of a lower load limit below which collapse will never occur is discussed. From a consideration of known creep behavior, it is concluded that there may be a temperature below which finite lower limits do exist and above which the lower limit is zero.

WADC TR 53-24 Pt 4  
ASTIA Document No. AD 118293  
OTS Release

May 1957

SUBJECT: INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT  
STRUCTURAL METALS

INVESTIGATOR: John Salvaggi

CONTRACT: AF 33(616)-2226

CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

ABSTRACT: Previous studies on the high-temperature creep and rupture behavior of a number of aircraft structural materials exposed to relatively simple intermittent-load or intermittent-heat test conditions emphasized the fact that each alloy system behaves in its own characteristic manner. Five of these materials, C-110M titanium, A-70 titanium, type 321 stainless steel, N-155 alloy, and 4130 steel were selected for further evaluation under the more complex conditions of combined intermittent temperature and load. Analysis of these test results disclosed little difference in creep and rupture behavior relative to the intermittent-load or intermittent-heat results.

In general, the basic mechanisms or causes responsible for the behavior patterns noted in the simpler environments of constant-temperature intermittent-load and constant-load intermittent-temperature are relatively unaffected by the phase relationships of the combined cyclic-load and temperature test conditions utilized in this study.

WADC TR 53-336 Pt 4  
OTS Release

May 1956

SUBJECT: THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS Part 4. Creep Results for Alclad 7075-T6 Aluminum Alloy and Comparison with Results for Other Materials

INVESTIGATOR: N. H. G. Daniels  
H. B. Masuda  
John E. Dorn

CONTRACT: AF 33(038)-11502

CONTRACTOR: University of California

ABSTRACT: A study was made of the creep properties of clad aluminum alloy 75S-T6 under intermittent heating and loading conditions at 300°F and 600°F. A few tests were also conducted at 450°F under intermittent heating conditions.

The results were correlated by means of the empirical functional equation

$$\epsilon = F \{ t f(\sigma) \}$$

where

$\epsilon$	=	total strain
$t$	=	time
$\sigma$	=	stress
$F$	=	strain function
$f$	=	stress function.

As suggested in previous reports, the function  $f$  was shown to be approximately independent of strain and of the type of intermittent testing cycle. The prediction of intermittent condition creep data can therefore be carried out from a knowledge of the constant load isothermal data, and a single test under the intermittent condition.

A review of the literature has shown that although in a significant proportion of cases the above analysis can be used successfully, the function  $f$  often varies considerably with strain, and in some instances is not independent of the type of intermittent condition. Nevertheless even the approximate prediction of intermittent creep data by flexible use of the method is likely to be of value.

The test results were also compared on a basis of net time at temperature under load. It appears that any intermittent cycle permitting recovery and overaging of the material in the absence of load has a deleterious effect on its creep resistance. Under other intermittent conditions the creep of the material is approximately the same on a net time basis as its creep under steady load isothermal conditions.

WADC TR 53-336 Pt 5  
OTS Release

May 1956

SUBJECT: THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS Part 5. Further Creep Results on Alclad 7075-T6 Aluminum Alloy and Consideration of Analytical Procedures

INVESTIGATOR: N. H. G. Daniels  
H. B. Masuda

CONTRACT: AF 33(038)-11502

CONTRACTOR: University of California

ABSTRACT: A further study of the creep properties of aluminum alloy Alclad 7075-T6 (Clad 75S-T6) under isothermal intermittent stressing and under cyclic temperature steady load conditions has been made at 300°F, 450°F, and 600°F.

For the intermittent stressing conditions, cycles in which the load was on for 5 minutes out of a 2 hour cycle, 5 minutes out of a 12 minute cycle, and 24 hours out of a 48 hour cycle were used. The first cycle was shown to have seriously deleterious effects, particularly at 300°F, where the net times under load to reach specified strains or fracture were reduced to approximately one eighth of the corresponding times for steady load isothermal creep.

At 300°F it was shown that the ratio of on-load time to off-load time controlled the scale of the effect of the intermittent condition, which was apparently independent of the total duration of each cycle. Empirical methods of analysis and prediction based on the equation used in previous work

$$\epsilon = F \{ t f(\sigma) \}$$

where  $\epsilon$  = total strain  
 $t$  = time  
 $\sigma$  = stress  
 $F$  = strain function  
 $f$  = stress function

were successfully applied to the results both at 300°F and 450°F.

Neither the above correlation method nor the use of comparisons based on net time under load were successful at 600°F for the extreme types of cycle used. Correlations for other alloys are also given.

The cyclic temperature, steady load creep data were also subjected to attempted analysis using other correlation methods available in the literature, but these did not seem superior to the above empirical method. Other intermittent condition creep data in the literature which have become available since the last report were also analyzed, and the limitations of the method, as discussed therein, were confirmed.

WADC TR 55-150 Pt 3  
ASTIA Document No. AD 97212  
OTS Release

August 1956

SUBJECT: MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS Part 3.  
Fatigue Evaluation of Magnesium Alloys  
INVESTIGATOR: W. S. Hyler  
F. H. Lyon  
CONTRACT: AF 33(616)-2303  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: WADC Technical Report 55-150, "Materials-Property-Design Criteria for Metals", indicated that the ANC-5 document shows discrepancies in fatigue data for various magnesium alloys as obtained in rotating-bending, plate-bending, and axial-loading fatigue tests. A study was made of three magnesium alloys FS-1a (AZ 31A-0), J-1 (AZ61A-F), and O-1 (AZ80A-F) under conditions of completely reversed stress for the three kinds of loading.

Results suggest that the discrepancies noted in ANC-5 data between rotating-beam fatigue data and data from the other two types of tests are real. Certain other inconsistencies in the data suggest that additional fatigue studies should be made to provide more reliable information.

A review of methods of presenting fatigue data in ANC-5 was made. It is suggested that a more consistent method of presentation be followed for the various alloy systems for which fatigue data are reported.

October 1956

SUBJECT: MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS Part 4.  
Elastic Moduli: Their Determination and Limits of Application

INVESTIGATOR: S. A. Gordon  
R. Simon  
W. P. Achbach

CONTRACT: AF 33(616)-2303

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A study was made of the modulus of elasticity at elevated temperatures for several materials as it is derived from the conventional stress-strain curve and as it is derived from the determination of the velocity of propagation of elastic waves. The two methods of determination give modulus values which agree closely in regions of low stress and where time effects are unimportant. At higher stress levels, where the stress-strain relationships are not linear and where time effects are important, moduli determined by the two methods do not agree, the dynamic modulus being higher than the statically determined modulus.

These differences determined for the magnesium alloy AZ-31 and for the aluminum alloy 2024-T4 for various temperatures are discussed with respect to their applicability to airframe design.

November 1956

SUBJECT: TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING

INVESTIGATOR: Joseph D. Morrison  
J. Robert Kattus

CONTRACT: AF 33(616)-424

CONTRACTOR: Southern Research Institute

ABSTRACT: The purpose of the work described in this report was to determine the effects of variations in strain rate and holding time on the tensile properties of several aircraft-structural metals after they had been heated within 10 seconds to test temperatures up to 1200°F. The work was primarily directed toward determining these effects on the yield and ultimate strength. Other properties such as modulus of elasticity, proportional limit and total elongation were also determined but only as by-products and for the purpose of establishing trends. These properties should be used only after careful consideration of the complex conditions under which they were obtained. The investigation covered strain rates from 0.00005 in./in./sec.

to 1.0 in./in./sec, holding times at test temperature from 10 seconds to 30 minutes, and the following materials:

1. Sheet metals: Annealed Stellite-25, precipitation-hardened Inconel-X half-hard and full-hard Type 301 stainless steel, 17-7PH stainless steel in both the annealed and the TH 1050 conditions, AISI-4130 steel in both the normalized and the quenched and tempered conditions, hot-rolled SAE-1020 steel, and Al10-AT and Ti-140A titanium alloys.
2. Cast metals: ZH-62T5 magnesium alloy and 356-T6 aluminum alloy.

With a few exceptions, the strength properties of the test metals increased appreciably with increasing strain rates, whereas percent elongation reacted erratically to changes in strain rate and modulus of elasticity was affected only slightly.

At certain temperatures and strain rates, variations in holding time significantly affected the strength and ductility of 356-T6 aluminum alloy and affected only to a minor degree the strength of Inconel-X, full-hard Type 301 stainless steel, both normalized and heat-treated AISI-4130 steel, and 17-7PH-TH1050 stainless steel. These effects are believed to be caused by structural changes. Variations in holding time had no effect on the properties of the other alloys.

Up to 1000°F, Type 17-7PH-TH1050 stainless steel was the strongest material tested, but above 1000°F precipitation-hardened Inconel-X was the strongest.

WADC TR 56-216  
ASTIA Document No. AD 110559  
OTS Release

November 1956

SUBJECT: THE PROPERTIES OF CONSTRUCTIONAL METALS AS A FUNCTION OF TEMPERATURE AND STRAIN RATE IN TORSION

INVESTIGATOR: E. P. Klier A. Viggiano  
N. Feola V. Weiss

CONTRACT: AF 33(616)-2606

CONTRACTOR: Syracuse University

ABSTRACT: Seven structural metals have been tested in torsion at four strain rates and at selected testing temperatures. The torque-twist relationships have been determined at the three lowest strain rates.

The experimental results confirm the trends established by Work and Dolan (1). These trends have been shown to arise from changes in the torque-twist diagrams for the respective materials in strain-rate intervals



which are essentially unchanged for the different metals and which center on a strain rate of about 0.1 in./in./sec.

Both solid and tubular specimens have been tested and for the tubular specimens the normal strain rate modification of the modulus of rupture is observed at high strain rates. Differences in the trends of the data for the solid and hollow specimens are attributed to the action of the heat sink which the core of the solid specimen constitutes.

It has been possible to explain the several types of properties vs. strain rate curves by means of alterations in the torque-twist curve arising from strain hardening, temperature increase in the specimen due to plastic working, and to strain rate modification of the yield strength.

WADC TR 56-395 Pt I  
ASTIA Document No. AD 110637  
OTS Release

December 1956

SUBJECT: DESIGN PROPERTIES OF HIGH STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS Part I. Dependence of Tension and Notch-Tension Properties of High-Strength Steels on a Number of Factors

INVESTIGATOR: B. B. Muvdi  
G. Sachs  
E. P. Klier

CONTRACT: AF 33(616)-2362

CONTRACTOR: Syracuse University

ABSTRACT: In this report are presented the results of tension and notch-tension tests performed on hot rolled sections from commercial, electric furnace heats of 4340, V-Mod. 4330, 98B40, Tricent (Inco), Super Hy-Tuf, Hy-Tuf and Super TM-2 steels. Tension tests were conducted on 0.28 in. dia. specimens. An exception was a single test completed on a smooth 0.9 in. dia. 4340 steel specimen in order to examine the effect of section size on the tension properties of this steel. Notch-tension tests were performed on 0.3, 0.5 and 0.9 in. dia. specimens that were heat treated to strength levels ranging between 180,000 and 300,000 psi approximately. These specimens were provided with notches leading to stress-concentration factors, K, of 3, 5 and 10. In both instances (tension and notch-tension tests) longitudinal and transverse specimens were examined. Furthermore, information from the literature pertaining to the effects of as-processed section size is considered and evaluated.

In general, the tensile strength was found to be independent of the specimen orientation, but to decrease gradually with increase in the specimen size. The ductility of smooth specimens, however, was observed to depend on both specimen orientation and specimen size.

The notch strength decreased with increase in stress concentration, specimen diameter and as-processed section size. It also decreased as the specimen orientation was changed from longitudinal to transverse. These effects were pronounced at high strength levels and diminished, with decrease in the tensile strength, to insignificant values at strength levels below 200,000 psi.

WADC TR 56-395 Pt II  
ASTIA Document No. AD 110619  
OTS Release

December 1956

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS Part II. Axial-Load Fatigue Properties of High-Strength Steels

INVESTIGATOR: B. B. Muvdi  
G. Sachs  
E. P. Klier

CONTRACT: AF 33(616)-2362

CONTRACTOR: Syracuse

ABSTRACT: In this report are presented the results of axial-load (completely reversed) fatigue tests on Tricent (Inco), Cru. SHS-260 and Super TM-2 steels heat treated to strength levels between approximately 250,000 and 300,000 psi. The fundamental effects of several variables on the fatigue properties of these steels are discussed and evaluated. These variables included the notch sharpness or the stress concentration, the strength level and the specimen orientation.

The results indicated that the fatigue strength was lowered as the stress-concentration factor was increased, a maximum lowering effect occurring for stress concentration factors between one and three. This effect was observed to depend upon the strength level. The endurance limit for both smooth and notched specimens developed minimum values at a strength level between 240,000 and 260,000 psi, and maximum values at a strength level between 270,000 and 300,000 psi approximately. In general, the endurance limit was found to be lower for transverse than for longitudinal specimens. Furthermore, this effect was much more pronounced for smooth than for notched specimens and was observed to be severe at high strength levels and to decrease with decrease in this quantity.

WADC TR 56-395 Pt III  
ASTIA Document No. AD 118167  
OTS Release

March 1957

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT Part III  
The Response of High-Strength Steels in the Range of 180,000-300,000 psi to Hydrogen Embrittlement from Cadmium Electroplating

WADC TR 53-373 Sup 4

38

INVESTIGATOR: E. P. Klier  
B. B. Muvdi  
G. Sachs  
CONTRACT: AF 33(616)-2362  
CONTRACTOR: Syracuse University  
ABSTRACT: The embrittlement of high strength steels due to the action of hydrogen introduced by Cd-electroplating has been studied in sustained-load, rotating beam fatigue, and bending tests. Strength levels from 180,000 to 300,000 psi as suitable for the various steels were examined for a variety of initial conditions of stress concentration.

All steels were found to be embrittled in some measure after Cd-plating and this embrittlement could not be fully eliminated, as determined in the bend test, through the baking treatment used. The improvement in properties which did result from baking was promoted by a redistribution and not an elimination of hydrogen from the steel.

Failure promoted by Cd-plating is affected by the experimental conditions and has been discussed at length in the report. In the hydrogen bearing zone a crack is initiated and then depending on the experimental conditions may propagate to failure of the cross section through overloading. Crack development is apparently dependent, in part, on the composition and is minimized by reduction in carbon content or by an increase in silicon content.

Both the sustained-load and bend tests are suitable tests for evaluation of hydrogen embrittlement in ultra-high strength steels. The rotating beam fatigue test is a relatively insensitive test of hydrogen embrittlement, but can be used to provide an excellent measure of the "static" notch strength of the steel.

#### ELECTRODEPOSITION

WADC TR 54-485 Pt III  
ASTIA Document No. AD 110402  
OTS Release

September 1956

SUBJECT: ELECTRODEPOSITION OF TITANIUM  
INVESTIGATOR: Walter E. Reid, Jr.  
Jean H. Connor  
Abner Brenner  
CONTRACT: AF 33(616)-53-11  
CONTRACTOR: National Bureau of Standards

**ABSTRACT:** The study of titanium-aluminum alloy baths as described in previous reports (1) was extended. The possibility of using sodium or potassium borohydride instead of lithium borohydride to prepare the titanium and zirconium borohydrides was examined. Among the new compounds prepared for use in the titanium-aluminum alloy bath were chloroborohydride etherates of titanium. Some work was done on the use of organometallic aluminum compounds in a titanium alloy bath rather than the hydride aluminum bath previously used. Alloys of titanium or zirconium with magnesium were obtained from a hydride type of bath. Codeposition of some metals similar to titanium, namely hafnium and thorium, with aluminum from the borohydride bath was studied for purposes of comparison with the titanium-aluminum alloys.

A number of reactions between reactive organic compounds and titanium salts were performed in the search for a suitable titanium plating bath. This included an unsuccessful attempt to prepare ethyltitanium and a tetraphenyl borate derivative of titanium.

### FATIGUE

WADC TN 55-273 Pt 2  
ASTIA Document No. AD 110491  
OTS Release

October 1956

**SUBJECT:** CUMULATIVE FATIGUE DAMAGE OF AIRCRAFT STRUCTURAL MATERIALS  
Part 2. 2024 and 7075 Aluminum Alloy Additional Data and Evaluation

**INVESTIGATOR:** Alfred M. Freudenthal  
Robert A. Heller

**CONTRACT:** AF 33(616)-2274

**CONTRACTOR:** Columbia University

**ABSTRACT:** The object of the investigation is to determine the effect of randomly varying stress-amplitudes representing gust or manoeuver load sequences encountered in flight on the fatigue life of 7075 and 2024 aluminum alloys by testing small rotating beam specimens subject to random time series of stress-amplitudes derived from specific frequency distributions of stress-amplitudes. The tests were carried out on specially built random fatigue testing machines of a type developed in the Civil Engineering Research Laboratories of Columbia University.

Most of the test results have been presented in Part 1 of the Technical Note. Some recent results are included in the present report. An approximate non-linear cumulative damage theory based on a thermal mechanism of fatigue supported by a purely statistical approach is developed. The theory approximates test results reasonably well for the unnotched specimens used in the investigation. Its validity, for specimens having stress concentrations, is currently being tested.

May 1956

SUBJECT: FATIGUE PROPERTIES OF VARIOUS GLASS-FIBER-REINFORCED PLASTIC LAMINATES  
INVESTIGATOR: K. H. Boller  
CONTRACT: DO 33(616)-54-14  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Fatigue strength values are presented for 6 standard and 4 heat-resistant resin laminates reinforced with glass fibers. Fifty-three S-N curves, representing fatigue data between 1 thousand and 10 million cycles, show the effect of fatigue strength of a notch, moisture, fabrics, resins, mean stress levels, angles to warp, and temperatures up to 500°F.

WADC TR 55-483  
ASTIA Document No. AD 97190  
OTS Release

August 1956

SUBJECT: STATISTICAL EVALUATION OF VARIATION IN ENDURANCE LIMIT AMONG SEVERAL HEATS OF PROPELLER TYPE STEEL  
INVESTIGATOR: W. L. Starkey  
S. M. Marco  
R. R. Gatts  
CONTRACT: AF 33(616)-2468  
CONTRACTOR: The Ohio State University Research Foundation  
ABSTRACT: Samples from three heats of SAE 4330, five heats of SAE 4340, and four heats of SAE 4350 aircraft quality steel were subjected to Prot-type rotating bending endurance tests. All specimens were heat treated to approximately Rockwell C 43 hardness. Separate estimates of the arithmetic average and the standard deviation of the endurance limit were determined for each heat from specimens selected parallel to the direction of mill rolling and from specimens selected perpendicular to that direction. An evaluation was made of the extent of agreement between the data obtained and a normal frequency distribution of the endurance limits of individual specimens. On the basis of normal distribution, confidence limits of 95% probability were established for each estimate of average endurance limit. The effects of various parameters on the average and on the standard deviation of specimen endurance limits were studied. These parameters included the carbon content, anisotropy, nonmetallic inclusion count, hardness, tensile strength, type of fatigue fracture, and origin of heat. Additional endurance tests were performed to compare the results of Prot-type tests with conventional endurance tests.

July 1956

SUBJECT: THE EFFECT OF STATIC MEAN STRESS ON THE DAMPING PROPERTIES OF MATERIALS

INVESTIGATOR: Neal L. Person  
Benjamin J. Lazan

CONTRACT: AF 33(616)-2803

CONTRACTOR: University of Minnesota

ABSTRACT: Vibrational stresses resulting from near-resonant operation are often encountered in current engineering practice under conditions which superimpose static mean stresses, such as, jet engine compressor and turbine blades. One method of minimizing vibration amplitude under near-resonant operating conditions is by the use of damping. Therefore, investigation of the damping properties of materials was undertaken.

New bending vibration decay equipment was developed to determine the effect of static mean stress on the damping associated with a given alternating stress. Tests were performed on SAE 1020 steel, 2024-T aluminum, J-1 magnesium, annealed RC-55 titanium, S-816 alloy, glass laminate plastic, and 403 stainless steel. In all cases the maximum stress on the test specimens was kept below the cyclic stress sensitivity limit, below which damping is unchanged by stress history.

403 was the only material that displayed a significant change in the damping due to the superimposed static mean stress. Whereas for the other materials the change was less than 30 percent, for 403 (a magneto-mechanical alloy) the specific damping energy decreased 90 percent when the mean stress was increased from zero to 40,000 psi.

WADC TR 56-37

March 1956

SUBJECT: DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF TITANIUM ALLOYS, HIGH TEMPERATURE ALLOYS, STAINLESS STEELS, AND GLASS LAMINATE AT ROOM AND ELEVATED TEMPERATURES

INVESTIGATOR: E. R. Podnieks  
B. J. Lazan

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: The significance of the damping, elasticity, and fatigue properties of a material on the resonant behavior of a part is reviewed. The basic unit for expressing damping energy and a procedure for its determination are discussed. Data on damping, elasticity, and fatigue properties at room and elevated temperatures are presented for four types of materials: titanium

alloys, high temperature alloys, stainless steels, and glass fabric laminate. The general behavior of these dynamic properties is summarized with regard to the significance of the more important variables. The resonant fatigue properties in form of resonant fatigue curves are determined for several characteristic types of parts by using the above materials. A comparison is made for the materials under different design criteria.

WADC TR 56-122  
ASTIA Document No. AD 97186  
OTS Release

August 1956

SUBJECT: EFFECT OF FATIGUE STRESS HISTORY ON ELASTICITY PROPERTIES AND STRESS DISTRIBUTION UNDER ROTATING BENDING  
INVESTIGATOR: Leonard C. Lidstrom  
Benjamin J. Lazan  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: Data are presented on the effect of fatigue stress amplitude and number of cycles on the stress-strain properties of mild steel under reversed axial (tension-compression) stress. Cyclic stress near the fatigue limit is shown to have a significant effect on the stress-strain properties and secant modulus of the material. The general significance of the observed changes in modulus properties on the stress distribution in a rotating beam are discussed.

An analytical method is presented for determining the actual or specific stress-strain relationships from the moment-strain data of rotating beam fatigue specimens. This method is used to calculate the specific stress-strain relationships for mild steel, and these are compared with experimentally determined axial stress-strain data. The agreement is found to be good.

The actual stress distributions in rotating cantilever-beam fatigue specimens are determined from the specific stress-strain relationships. The errors associated with nominal stress based on the accepted linear stress-strain relationship are analyzed. The effects of magnitude of stress and number of stress cycles on stress distribution are discussed. Fatigue data are presented on solid and hollow rotating beams and under axial stress. These data, compared on the basis of nominal stress, show poor agreement (12 to 30%). However, if compared on the basis of specific or true stress, determined by the methods presented, the agreement is reasonably good (3 - 7%).

WADC TR 56-127  
ASTIA Document No. AD 110492  
OTS Release

October 1956

SUBJECT: EFFECT OF CHANGING CYCLIC MODULUS ON BENDING FATIGUE STRENGTH  
INVESTIGATOR: A. A. Blatherwick  
B. J. Lazan  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: The implications of changing cyclic properties of a material are analyzed by means of hypothetical cyclic stress-strain curves. The effects of cross-sectional shape of specimen and of loading conditions on fatigue strength are investigated through calculations based on the assumed material behavior. Two types of material are considered, one of which has a strain-hardening characteristic, while the other becomes softer under repeated cycling. In the second phase of the work, actual materials are tested in reversed-flexure fatigue to provide experimental verification of the results obtained in the hypothetical analysis. It is concluded that the type of material is exceedingly important in determining the effect of specimen shape and type of test on fatigue strength.

WADC TR 56-180  
OTS Release

June 1956

SUBJECT: BIBLIOGRAPHY OF THE MATERIAL DAMPING FIELD (With Abstracts and Punched Card Codings)  
INVESTIGATOR: L. J. Demer  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: This bibliography was compiled as an aid in furthering current and future research in the field of the damping of materials and structures. Consisting of almost 900 entries, it is believed to be the most extensive such collection presently available. An abstract of each reference is included in all but a few cases. A detailed classification system for the Damping Field is described which differs from the ASM-SLA Metallurgical Literature Classification only in the Processes and Property Index. Directions for use of the Damping Field classification system and ASM-SLA punched filing cards in conjunction with the present bibliography are given. Punched card codings are included with each reference in the bibliography so that the entries in this report copy may be used in constructing a personal punched card filing system of the damping field for the individual user.

WADC TR 56-181  
ASTIA Document No. AD 97240  
OTS Release

August 1956

SUBJECT: FATIGUE, CREEP, AND RUPTURE PROPERTIES OF HEAT RESISTANT MATERIALS  
INVESTIGATOR: F. H. Vitovec  
B. J. Lazan

WADC TR 53-373 Sup 4



CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: Fatigue, rupture, and creep data at various temperatures obtained under various combinations of mean and alternating stress are presented for the alloys Stellite 31, S-816, 6.3% Mo-Waspalloy, 7% Mo-Waspalloy, M-252, Inconel X-550, 16-25-6 Timken, Crucible 422, Lapelloy and Stainless type 403. Tests were performed under axial stress on unnotched specimens and specimens having theoretical stress concentration factors of 2.4 and 3.4. The data are presented as S-N curves and stress range diagrams to show the effect on the fatigue and creep properties of specimen notch, temperature, ratio of alternating-to-mean stress, and stress magnitude. The role of both creep and fatigue as design factors and the relation of notch sensitivity to loading conditions are discussed with particular reference to temperature and ratio of alternating-to-mean stress.

WADC TR 56-289  
ASTIA Document No. AD 97211  
OTS Release

August 1956

SUBJECT: EFFECTS OF SPECIMEN PREPARATION ON FATIGUE  
INVESTIGATOR: Franz H. Vitovec  
Harold F. Binder  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: The effect of surface preparation methods for plain specimens on the fatigue strength is reviewed. The various effects caused by machining, grinding and mechanical and electrolytical polishing are analyzed. A study of the penetration of plastic deformation caused by the notch preparation is presented. Data on the effect of lapping procedure on the Prot failure stress of SAE B1113 steel and SAE 1020 steel are reported. The direct stress fatigue properties of notched specimens from these two steels were not affected by the direction of lapping

WADC TR 56-296  
ASTIA Document No. AD 97254  
OTS Release

September 1956

SUBJECT: NUCLEAR RADIATION OF REINFORCED PLASTIC RADOME MATERIALS  
INVESTIGATOR: Robert C. Tomashot  
Douglas G. Harvey, 1/Lt  
ABSTRACT: Nine different reinforced plastic laminate materials, each made with a different laminating resin, and one alkyd-isocyanate foam core-glass fabric faced sandwich material were subjected to integrated gamma radiation dosages up to 109 roentgens. After completion of the radiation exposures, the materials were tested to determine the flexural, tensile, and compression strengths under both standard and wet conditions. The mechanical properties of the heat resistant plastic laminates were also determined at elevated temperature.

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Dielectric constant and loss tangent measurements were conducted on irradiated samples to determine the effect of radiation on the electrical properties of these materials.

Data obtained from these tests show that the mechanical properties were not significantly affected except for one epoxy type resin laminate. None of the materials showed any significant change in electrical properties due to radiation.

WADC TR 56-408  
ASTIA Document No. AD 118157  
OTS Release

March 1957

SUBJECT: INTERRELATION OF FATIGUE CRACKING DAMPING AND NOTCH SENSITIVITY  
INVESTIGATOR: L. J. Demer  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: The materials tested in this study were the heat resistant alloy N-155 a high carbon steel in two conditions of heat treatment, gray iron, aluminum alloy, J-1 magnesium alloy, and SAE 1020 steel. The number of cycles to initiation of macrocracking and to fracture were determined for both unnotched and notched specimens during fatigue tests performed over a wide range of stress levels. Crack detection was principally by the moist coating and deflection methods. Other supplementary techniques were also employed. The damping and stiffness behaviors of the specimens were observed during the fatigue tests to determine the characteristic changes taking place at the higher stress levels both prior to, and following the initiation of cracking.

Comparisons of the damping and stiffness properties of the specimens prior to cracking are made to determine the correlations existing with the static properties of the materials. Analyses are also made of the tendency toward ease of crack initiation and also of the rates of crack growth in the various materials. The dependence of these properties on the type of material is shown. Relationships are indicated between the damping and fatigue strength properties of both unnotched and notched specimen types. Variations of the fatigue strength reduction factors with number of stress cycles are determined both for fracture data and also for crack initiation data. A possible relation is indicated between the damping and the stiffness properties and the notch sensitivity properties of the materials. In addition, observations are presented on the cracking and fracture behavior of unnotched and notched specimens of the materials tested and the variations in these characteristics with stress level of the tests.

FUR

JOINING

WADC TR 52-322 Pt 4

November 1955

SUBJECT: CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS  
INVESTIGATOR: A. L. Lowe, Jr.  
R. P. Sopher  
P. J. Rieppel  
CONTRACT: AF 33(616)-2734  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report summarizes the experimental work conducted at Battelle in a study to determine the causes of cracking in high-strength weld metals. A new hot-tension machine was designed and built to facilitate the testing of SAE 4340 weld metals on cooling from the molten state. The tests were conducted over the temperature range from 2600°F to 100°F. Results from the studies showed phosphorus to be detrimental to weld-metal cracking resistance. As the phosphorus was increased the ductility was lowered at temperatures near the solidus. Nitrogen content within the normal range of SAE 4340 steels appeared to have little influence on the cracking resistance of the weld deposits. Rare-earth metal additions improved the hot ductility and hot-cracking resistance of the weld metals. An increase in silicon lowered the hot ductility and hot-cracking resistance slightly.

WADC TR 54-17 Pt 3  
ASTIA Document No. AD 110570  
OTS Release

November 1956

SUBJECT: JOINING OF MOLYBDENUM  
INVESTIGATOR: William N. Platte  
CONTRACT: AF 18(600)-114  
CONTRACTOR: Westinghouse Research Laboratories  
ABSTRACT: The use of welding atmospheres of purified argon and helium is shown to cause very little contamination of the weld metal in molybdenum fusion welds. However, the use of high purity atmospheres provides only a small improvement in ductility over welds made in welding grade argon.

Increasing the carbon content of the base material within the range 0.04% to 0.06% appears to improve the ductility of the weld metal. Additions of 0.5% titanium in the presence of 0.05% carbon gave welds which could be deflected 120° at 80°F. Increasing the carbon content from 0.015 to 0.05% improved the ductility of the molybdenum welds containing 0.5% titanium. Additions of 0.16% aluminum to the molybdenum yielded welds which could be deflected 120° at 120°F.

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Exploratory studies on the effectiveness of diffusion as a method of removal of nitrogen from the weld metal are discussed. The use of thermal treatment as a method of improving mechanical properties by over-aging and polygonization was explored. Weld metal additions are also discussed briefly. The use of Ti and Zr as deoxidizers in vacuum sintered molybdenum is described.

An exploration into the effects of residual welding stresses showed that the weld crater cracking encountered in many cases is caused by measurable stresses in the region of the weld which approximate the rupture strength in material.

WADC TR 56-550  
ASTIA Document No. AD 118210  
OTS Release

April 1957

SUBJECT: DEVELOPMENT OF HIGH-STRENGTH FILLER WIRES FOR WELDING SAE 4130, 4140, AND 4340 STEELS  
INVESTIGATOR: Herbert W. Mishler  
Raeman P. Sopher  
CONTRACT: AF 33(616)-2339  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Filler wires were developed for welding high-strength low-alloy aircraft steels by the inert-gas tungsten-arc and inert-gas consumable-electrode processes.

The major factors that were considered in the evaluation and development work were: (1) weld-metal porosity, (2) weld-metal cracking tendency, (3) heat-treatment response compared with base metal, (4) strength of heat-treated weld joint, (5) weld-metal toughness, and (6) weld-metal hardenability. Two filler wires were developed for welding each of the three steels, SAE 4130, 4140, and 4340. One of each pair of filler wires had a composition similar to the base material, while the other was of a dissimilar composition. Sound, crack-free welds, heat treatable to 200,000 psi were deposited under a high degree of restraint with all these filler wires. The best results were obtained with the filler wires of compositions similar to the base material, although satisfactory welds were made also at most of the strength levels with the dissimilar filler wires.

LEATHER

## METALS

### General

WADC TR 54-472 Pt 2  
OTS Release

May 1956

SUBJECT: DEVELOPMENT OF AUSTENITIC IRON-BASE SHEET ALLOY  
INVESTIGATOR: Roy R. Rothermel  
CONTRACT: AF 33(616)-2047  
CONTRACTOR: Crucible Steel Company of America  
ABSTRACT: Results of the investigation of the weldability of a type II, AD30 alloy (conducted in the welding laboratory of the contractor) indicate that this material can be welded when adequate measures are taken to limit stresses set up during welding and where proper protection from the atmosphere is afforded the material during welding.

Satisfactory welds were made in representative types of weld joints by the metallic arc process using types 310 and 312 mod. (2-3%Mo) electrodes and by the inert gas process using AD30 filler.

Satisfactory welds were obtained by the butt flashwelding process.

Bend test results of various welded specimens show good ductility, an evidence of formability of a welded section in the as-welded, solution treated condition.

Short time tensile test results indicate welded specimens to have strengths (at a 1500°F testing temperature) comparable to the AD30 base material.

WADC TR 56-83  
OTS Release

April 1956

SUBJECT: AN INVESTIGATION OF THE EFFECTS OF HYDROGEN ON THE BRITTLE FAILURE OF HIGH-STRENGTH STEELS  
INVESTIGATOR: Edward R. Slaughter                      Arthur R. Elsea  
                  E. Ellis Fletcher                      George K. Manning  
CONTRACT: AF 33(616)-2103  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: The effect of hydrogen on the delayed, brittle failure of high-strength steel was investigated by the use of room-temperature stress-rupture tests on unnotched, cathodically charged specimens. The principal variables in these tests were stress, strength level, structure, composition, and hydrogen

content. The time for failure to occur decreased as the strength level of the steel or the applied stress was increased, but changes in structure or composition had little effect on the time to failure.

The martensite and bainite reactions were found to be unaffected by the variations in hydrogen content.

The diffusion of hydrogen through cathodically charged steel was investigated. In this study, stress was found to have no measurable effect on either the diffusion or the distribution of hydrogen in steel.

Hydrogen contents of steel cathodically charged under various conditions were determined.

WADC TR 56-242  
ASTIA Document No. AD 97215  
OTS Release

August 1956

SUBJECT: STRESS CORROSION CRACKING IN TYPE 403 STAINLESS STEEL  
INVESTIGATOR: Mars G. Fontana  
CONTRACT: AF 33(616)-2849  
CONTRACTOR: The Ohio State University Research Foundation  
ABSTRACT: The effect of austenitizing temperature on the hardness, impact strength, and microstructure of Types 403, 420, and 431 stainless steel was determined. The optimum combination of these properties occurred with austenitizing temperatures of 1725°, 1850° and 1900°F respectively for the 403, 420, and 431 materials. Tempered structures displayed minimums in the impact strength, tempering temperature curves at 1000°, 900 and 1000°F for the 403, 420 and 431 materials respectively.

Metallographic studies were made of stress-corrosion specimens tested in a 1:1 HCl and water solution containing 1% SeO<sub>2</sub>. These studies showed that pitting was initiated at MnS inclusions and that cracking was associated with the pits. Also, pitting characteristics were dependent on tempering temperatures and therefore are related to the microstructure of the alloy. Cracking does not occur in the Type 403 alloy when it is tempered at 1050°F or higher. This temperature is lowered to 900°F for the Type 420 material, and it is less than 700°F for Type 431 stainless steel stressed to 75,000 psi and tested in the above solution.

Electron microscopy and diffraction studies were made of tempered Type 403 stainless steel. These studies indicate several carbides to be present in the alloy after tempering in the 1000°-1200°F temperature range. Diffraction patterns vary for specimens tempered in this range.

August 1956

SUBJECT: THE EFFECT OF STRESS ON MICROHARDNESS  
INVESTIGATOR: F. H. Vitovec  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: Microhardness is investigated as a function of test load for mild steel, Armco iron, copper, brass, zinc and cadmium. At light loads a discontinuity of slope in the hardness-load curve is observed which can be attributed to subgrain structure. A tension-compression fixture was developed and used to study the effect of stress on the microhardness of mild steel, brass, and copper specimens. For initially stress free specimens, tensile stress causes a decrease in microhardness over a certain range of test load, and compressive stress results in a slight increase. The effect of stress on microhardness is complicated by the discontinuous slope in the hardness-load curve introduced by subgrain structure effects.

WADC TR 56-294  
ASTIA Document No. AD 97235  
OTS Release

August 1956

SUBJECT: RETAINER MATERIALS FOR AIRCRAFT GAS-TURBINE BEARINGS  
INVESTIGATOR: Peter F. Mataich  
F. Clifton Wagner  
CONTRACT: AF 33(616)-2949  
CONTRACTOR: Horizons Incorporated  
ABSTRACT: This study was made with the purpose of developing new cage materials to be used in gas-turbine jet engines. The materials being used presently are operating near their upper temperature limits and the demands for greater efficiency and power will necessitate the use of materials with high temperature wear characteristics superior to those presently being used.

The development work was concentrated on nickel base silver impregnated materials produced by powder metallurgy techniques. Various boride, carbide, and silicide additions were made to the nickel base, and several compositions were found which had superior wear characteristics. In addition to this work a series of oil evaluation tests were made, and the wear rates of both the new and the standard materials were determined while using these oils as lubricants.

WADC TR 56-330 Pt I  
ASTIA Document No. AD 118178  
OTS Release

April 1957

SUBJECT: THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS Part I. Eccentrically-Loaded Tension Members Having Angle- and T-Sections

INVESTIGATOR: O. M. Sidebottom  
M. E. Clark

CONTRACT: AF 33(616)-2753

CONTRACTOR: University of Illinois

ABSTRACT: This paper presents the results of an analytical and experimental investigation for the determination of the load necessary to produce any given depth of yielding in angle- and T-section members subjected to eccentric tensile loading. This load is found analytically from the intersection of a constant depth of yielding interaction curve and a moment-load curve. General load and moment expressions used in the construction of the interaction curves were derived for cross-sections composed of rectangular elements. The primary assumption in these derivations was that for small inelastic strains the stress-strain diagrams of the material could be represented by two straight lines. The moment-load expressions were derived on the assumption that each eccentrically loaded member deformed into a segment of a circle.

In the experimental investigation eccentric load tests were made on angle- and T-section members made from various classes of materials. The three materials used were S.A.E. 4340 alloy steel, type 304 stainless steel, and 7075-T6 aluminum alloy. The angle-section members were tested with the principal axes orientated in three different positions with respect to the loading plane. In general the results of these tests corroborated the theoretical analyses. It was found that deviations from the theory could be explained by variations in the stress-strain properties of the material.

Supplementary tests were performed to determine the effect of pure bending loading and the effect of time on the inelastic behavior under sustained and alternating load schedules. Pure bending test results indicated that the theory proposed was conservative in the majority of cases. The results of the sustained and alternating load tests indicated that time was not an important variable so long as the material properties used in the analysis were obtained from specimens subjected to loading schedules similar to those used in testing the eccentrically loaded member.

WADC TR 56-330 Pt II  
ASTIA Document No. AD 118185  
OTS Release

April 1957

SUBJECT: THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS Part II. Inelastic Behavior of Aluminum Alloy I-Beams with Rectangular Web Section Cutouts



INVESTIGATOR: Will J. Worley  
Shuji Taira  
CONTRACT: AF 33(616)-2753  
CONTRACTOR: University of Illinois  
ABSTRACT: This report contains an exploratory investigation of the plastic bending of aluminum alloy I-beams with rectangular web section cutouts. The mechanism method of analysis employing the upper bound theorem was employed in predicting the ultimate loads of the various beams. The experimental results are in good agreement with the predicted ultimate loads.

WADC TR 56-402  
ASTIA Document No. AD 110436  
OTS Release

October 1956

SUBJECT: RESEARCH ON EFFECTS OF PRETRAINING AND NOTCH SHARPNESS ON THE NOTCH STRENGTH OF MATERIALS  
INVESTIGATOR: G. W. Geil  
N. L. Carwile  
CONTRACT: AF 33(616)-55-9  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: A general study was made on the effect of prestraining in tension at +25°C on the tensile properties at -196° and +100°C of notched and unnotched specimens of (1) a commercially pure titanium, Ti-75A, and (2) a 4% Al, 4% Mn titanium alloy, C130AM. Included in this general study were (1) a preliminary investigation on both materials to determine the effect of the notch sharpness, as varied by a change in root radius or notch depth, on the tensile properties, (2) a study of the combined effect of notch sharpness (as varied by a change in notch depth) and prestrain at +25°C on the tensile properties of notched specimens of these materials at -196° or +100°C, and (3) a determination of the impact properties of Charpy V-notch specimens of both metals over the temperature range of -196° to +300°C.

Prestraining in tension at +25°C had considerable effect on the ductility retained by both the notched and unnotched specimens of each material at -196° or +100°C. Furthermore, the retained true strain at initial fracture (total true strain at initial fracture in the second stage of test at -196° or +100°C minus the true strain at the end of the first stage of test at +25°C) decreased with increase in the prestrain at +25°C. The results indicate that Ti-75A and C130AM are not subject to "rheotropic embrittlement" as the retained ductility of the specimens at -196°C was not improved by prestraining at +25°C.

Prestraining in tension at +25°C had little, if any effect on the tensile strength or notch strength of Ti-75A or C130AM, except for some impairment in the notch strength of the deep-notched specimens of C130AM that did not retain any appreciable ductility at -196°C.

The change from a ductile to brittle behavior in the impact tests on Charpy V-notch specimens (based on the energy absorbed in fracturing the specimens) occurred over temperature ranges of approximately +150° to +100°C and +200° to +100°C for Ti-75A and Cl30AM, respectively.

WADC TR 56-486  
ASTIA Document No. AD 110582  
OTS Release

November 1956

SUBJECT: EVALUATION OF POROUS MATERIALS FOR BOUNDARY-LAYER CONTROL  
INVESTIGATOR: David E. Debeau  
CONTRACT: AF 33(600)-28835  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Criteria were determined from designers for comparing various commercial permeable sheet materials for use in boundary-layer control associated with high-lift systems. Sintered metal powders, special woven and sintered wire materials, a compressed glass-fiber product, woven wire cloths, and perforated metal sheets were evaluated and compared for the following properties: average permeability, uniformity of permeability, resistance to clogging and corrosion, mechanical properties including room temperature tensile strength, modulus of elasticity, Poisson's ratio, and minimum bend radius. An evaluation of economic factors, including production facilities, product limitations, requirements for and availability of raw materials, and cost of product, was carried out on those permeable sheet materials which attained most nearly the physical and mechanical properties desired by the aircraft designers.

#### Ferrous

WADC TR 56-396  
ASTIA Document No. AD 110638  
OTS Release

December 1956

SUBJECT: TEMPER BRITTLENESS OF BORON-TREATED STEEL  
INVESTIGATOR: Samuel J. Rosenberg  
CONTRACT: AF 33(616)-55-8  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: Two series of steels, melted to the base composition of 8140, were studied to ascertain whether titanium and zirconium (present in many commercial boron addition agents) had any adverse effect upon the impact properties of the base steels, particularly with reference to temper brittleness. All steels were given the same hardening treatment (1/2 hour at 1600°F, oil quench) and were tempered 1 hour at 1200°F. The rate of cooling from the tempering temperature was varied so as to develop temper brittleness and isothermal embrittling treatments also were employed.

High Temperature

WADC TR 54-175 Pt 3  
ASTIA Document No. AD 97253  
OTS Release

September 1956

SUBJECT: NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURES Part 3. Final Data and Correlations  
INVESTIGATOR: Howard R. Voorhees  
James W. Freeman  
CONTRACT: AF 18(600)-62  
CONTRACTOR: University of Michigan  
ABSTRACT: Earlier parts of this report summarized rupture lives for smooth and notched round specimens of three heat-resistant alloys, together with pertinent experimental data on tensile stress-strain properties and creep-relaxation characteristics for these alloys. Similar types of data have now been obtained for flat specimens, and for two other types of material (a Cr-Si-Mo-V steel and an age-hardening aluminum alloy).

Results gathered during the over-all program lead to the following conclusions:

1. Elevated-temperature rupture characteristics of notched specimens under a steady tensile load appear to depend on three major factors:
  - a. The distribution and level of the initial stress pattern, determined by the notch configuration and tensile characteristics of the alloy.
  - b. The rate at which variable creep rates at different locations in the cross section are able to relax the peak stress originally concentrated near the notch. Under multiaxial stressing the effective stress can easily become less than the nominal value for alloys with low creep resistance.
  - c. Rupture characteristics of the material at the prevailing stresses and for the prior history experienced by different fibers in the notched bar. If too large a portion of the total life is used up at the initial high stresses, the remaining service will be short even if the final stress is low.

2. It appears that rupture life of a structure in the presence of a concentrated complex stress involves no new factors beyond those which determine rupture under simple stress. If one can predict the stress-strain-time history at points throughout a body, the time until rupture at any point seems amenable to calculation from data obtained with smooth specimens. The major uncertainty is choice of the proper criterion for initiation of rupture following creep under variable complex stresses.

3. For some alloys a small amount of plastic deformation near the notch when the load is added may radically alter subsequent creep-rupture characteristics. This may be the major factor in notch weakening under some conditions.

WADC TR 54-270 Pt I

June 1957

SUBJECT: INVESTIGATION OF THE COMPRESSIVE, BEARING AND SHEAR CREEP-  
RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS  
AT ELEVATED TEMPERATURES

INVESTIGATOR: F. J. Vawter L. A. Yerkovich  
G. J. Guarnieri G. Derrick

CONTRACT: AF 33(616)-190

CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

ABSTRACT: The intent of this investigation is to supplement conventional tensile creep data of several aircraft structural alloys with compression, bearing, and shear properties as well. While these data alone are of interest, a correlation is being attempted between tensile creep and compression, bearing, and shear creep properties so that the latter type of data may be predicted from tensile creep properties alone.

This report includes descriptions of equipment and fixtures for conducting tensile, compression, bearing, and shear creep tests. Tensile creep properties are reported at several test temperatures for the following alloys: (1) 2024-T3 aluminum sheet, 0.064 and 3/16 inch thick; (2) C-110M titanium sheet; (3) type 321 stainless steel sheet; (4) 2117-T4 aluminum rivet wire; (5) Monel rivet wire; and (6) type 301 stainless steel rivet wire. Bearing and shear creep characteristics are included for the 2024-T3 aluminum alloy.

WADC TR 54-270 Pt 2  
ASTIA Document No. AD 97283  
OTS Release

September 1956

SUBJECT: INVESTIGATION OF THE COMPRESSIVE, BEARING, AND SHEAR CREEP-  
RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS  
AT ELEVATED TEMPERATURES

WADC TR 53-373 Sup 4

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INVESTIGATOR: Frank J. Vawter Luke A. Yerkovich  
Glen J. Guarnieri George Derrick  
CONTRACT: AF 33(616)-190  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.  
ABSTRACT: The establishment of high-temperature creep and rupture properties of materials is a prerequisite for design if exposure to elevated temperature in service is anticipated. These properties, which are determined from the conventional tensile creep test, are of questionable value if stress conditions other than tension are encountered. To supplement these existing tensile creep and rupture data, this project was initiated to determine the high-temperature creep strengths of a number of structural aircraft alloys when subjected to compression, bearing, and shear stresses.

Tensile creep data are included for A-70 commercially-pure titanium, C-110M titanium alloy and SAE 4130 alloy steel sheet. Bearing creep data are presented for A-70 and C-110M titanium, 4130 steel and type 321 stainless steel. In addition, results of shear-pin deformation tests on 2117-T4 aluminum, Monel and type 301 stainless steel wire and compression creep test results for 2024-T3 aluminum sheet and plate and C-110M titanium alloy sheet are included.

WADC TN 55-290 Pt II  
ASTIA Document No. AD 110716  
OTS Release

January 1957

SUBJECT: DEVELOPMENT OF CAST IRON-BASE ALLOYS OF AUSTENITIC TYPE FOR HIGH HEAT-RESISTANCE AND SCALE-RESISTANCE  
INVESTIGATOR: F. Eberle  
J. H. Hoke  
W. E. Leyda  
CONTRACT: AF 33(616)-2413  
CONTRACTOR: The Babcock & Wilcox Company Research Center  
ABSTRACT: Iron-base alloys containing a minimum iron content of 45%, a minimum carbon content of 0.50%, a chromium content of not less than 20%, and between 27 and 32% nickel, part of which may be replaced by cobalt, can develop a creep-rupture strength at 1600° to 1800°F approaching that of the cobalt-base alloy H. S. 21 when they are strengthened by not more than 5% additions of Cb, Ta, Mo, and W in certain combinations and relative concentrations. These high-creep-rupture-strength iron-base alloys are equal to H. S. 21 in oxidation resistance at 2000°F, but inferior to the former in short-time cold and hot tensile strength, in cold ductility, and in resistance to thermal shock and thermal fatigue. The possibility of improving the latter by lowering the carbon content and compensating the resulting decrease in rupture strength with suitable modifications in composition has not been explored.

December 1955

SUBJECT: TENSILE, FRACTURE, AND SHORT-TIME CREEP PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING

INVESTIGATOR: J. Robert Kattus  
Clifford L. Dotson

CONTRACT: AF 33(616)-2837

CONTRACTOR: Southern Research Institute

ABSTRACT: The tensile, fracture, and short-time creep properties of electrolytic copper sheet, ingot iron sheet, tantalum sheet, molybdenum sheet, and four types of graphite - types GBE, 3474-D, 7087, and GBH - were determined at temperatures up to the melting points of the metals and up to 5200°F for the graphite. High rates of heating were used in all tests, and the tensile tests were conducted at moderate to rapid strain rates after relatively short holding times at the test temperatures.

The metals decreased in tensile strength continuously with increasing temperatures, but they did retain some strength at temperatures approaching the melting points. The graphite materials decreased little in strength between room temperature and 5200°F.

The copper and iron were capable of supporting a stress of 200 psi for five minutes at temperatures near the melting points. For five minutes at temperatures between 4600°F and 4800°F, each type of graphite supported a load equivalent to two-thirds of the room-temperature strength. Because of deterioration of the specimens due to oxidation, molybdenum and tantalum were not able to support a stress of 200 psi for as long as one minute at temperatures near their melting points. At maximum temperatures of 1900°F and 2200°F, the molybdenum and tantalum, respectively, were capable of supporting a stress of 10,000 psi for five minutes.

WADC TR 55-470 Pt 2  
ASTIA Document No. AD 110440  
OTS Release

October 1956

SUBJECT: AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS

INVESTIGATOR: Clarence A. Siebert  
Maurice J. Sinnott

Lynn H. DeSmyter  
Harry M. Ferrari

CONTRACT: AF 33(616)-353

CONTRACTOR: University of Michigan

ABSTRACT: Specimens from Chromel ASM, Hastelloy B, and commercial and vacuum-melted type 310 stainless steel alloys were oxidized for 100-hour periods in the stressed condition. Intergranular oxidation measurements were obtained

microscopically. In general, the intergranular penetrations increased rapidly with stress after a certain minimum value was reached. This minimum value, denoted as the threshold stress, was determined for each alloy at various temperatures. The weight gained during oxidation was determined. It was found that most of the alloys tested followed the parabolic oxidation law.

WADC TR 55-501  
OTS Release

March 1956

SUBJECT: DEVELOPMENT OF HEAT RESISTANT ALLOYS BY POWDER METALLURGY  
TECHNIQUES  
INVESTIGATOR: G. Zuremsky H. S. Kalish  
L. Sama L. L. Seigle  
CONTRACT: AF 33(616)-2401  
CONTRACTOR: Sylvania Electric Products, Inc.  
ABSTRACT: Powder metallurgical techniques were utilized in the production of Cr-W-Co alloys, to meet a stress rupture specification of 100 hours life at 870°C and 25,000 psi. Attempts to produce chromium-base alloys with room temperature ductility and/or high density were unsuccessful. The use of a master alloy improved the densities of the cobalt-base alloys. Stress-rupture properties of the cobalt-base alloys at 870°C and 25,000 psi appeared to approach those of the weaker Stellites but failed to meet the 100-hour requirement. Nickel additions lowered the stress-rupture strength but gave increased ductility. Attempts to retain carbon as an alloying addition were unsuccessful.

WADC TR 56-114  
OTS Release

April 1956

SUBJECT: INVESTIGATION OF THE EFFECTS OF INCONGRUOUS ELEMENTS AND THE INTERACTION EFFECTS OF THESE ELEMENTS ON HIGH TEMPERATURE STRENGTH OF FE-CO-NI-CR ALLOYS  
INVESTIGATOR: Thomas L. Robertshaw  
Francis M. Richmond  
CONTRACT: AF 33(616)-2777  
CONTRACTOR: Universal-Cyclops Steel Corporation  
ABSTRACT: An investigation was made of the effect of various hardening elements on the high temperature properties of vacuum melted alloys having a base composition of 60 atomic % Ni, 20 atomic % Cr, 10 atomic % Fe and 10 atomic % Co. Stress-rupture properties of these alloys at 1600°F were largely dependent on (1) the vacuum obtained during melting, (2) the fabrication methods, and (3) the total amount of hardener. On the basis of atomic misfit, the hardening elements B, Zr and Cb were equivalent to Mo in increasing the rupture strength of these alloys. On this same basis, however, Ti was more potent and Al less potent than Mo. A maximum in stress-rupture stress occurred at a hardener level of about 35 equivalent atomic percent Mo.

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It was found that, in general, a high level of strength was more likely to occur at a given hardener level when greater variety of hardening elements were added.

WADC TR 56-190  
OTS Release

May 1956

SUBJECT: A STUDY OF THE POSSIBILITY OF REINFORCING HIGH-TEMPERATURE ALLOYS BY ADDITION OF REFRACTORY POWDERS  
INVESTIGATOR: John D. Burney  
CONTRACT: AF 33(616)-2959  
CONTRACTOR: P. R. Mallory & Co., Inc.  
ABSTRACT: A study of the possibility of reinforcing 80 Ni - 20 Cr alloy by the addition of such refractory oxides as  $Al_2O_3$ ,  $TiO_2$ ,  $ZrO_2$ ,  $Cr_2O_3$  and  $SiO_2$  was made.

Several powder metallurgical fabrication techniques were investigated such as (1) pressing and sintering, (2) pressing, sintering and repressing, (3) pressing, sintering followed by hot working, (4) hot pressing and (5) liquid phase sintering.

In the "press-sinter" method of fabrication, bars with 1.0%  $TiO_2$  showed better stress-rupture properties than bars with any of the other oxide additions investigated. Rupture times were 7.45 and 8.7 hours at 1600°F and 7000 psi.

Hot forging techniques were found to be applicable for working bars containing oxide additions of 1.0% or less.

An 80 Ni - 20 Cr alloy reinforced with 1%  $Al_2O_3$  and densified by a liquid phase sintering technique resulted in improved stress-rupture properties compared to wrought 80 Ni - 20 Cr alloy. This material had a rupture life at 1600°F and 7000 psi in excess of 268 hours; while at 1800°F and 6650 psi, the rupture life was almost 47 hours.

The room temperature impact and ultimate tensile strength appeared adequate and the oxidation resistance and thermal shock properties were good.

WADC TR 56-327  
ASTIA Document No. AD 110409  
OTS Release

October 1956

SUBJECT: INVESTIGATION OF FORGED COBALT BASE ALLOYS FOR HIGH TEMPERATURE APPLICATIONS

WADC TR 53-373 Sup 4

60



INVESTIGATOR: R. R. MacFarlane  
R. K. Pitler  
E. E. Reynolds  
CONTRACT: AF 33(616)-2882  
CONTRACTOR: Allegheny Ludlum Steel Corporation  
ABSTRACT: Developmental studies were made of a wrought Co-base alloy for applications at high temperatures. Additions of Al improved oxidation resistance and lowered rupture strength. B additions up to .2 percent increased rupture strength and ductility. This increase was limited by a necessarily low solution temperature of 2150°F since, at 2200°F, eutectic melting of a B phase occurred. Ti additions resulted in a large increase in room temperature hardness through an aging reaction but yielded no improvement otherwise. Attempts at improving the workability of unforgeable air melted modifications by vacuum consumable electrode and vacuum induction melting techniques were not successful.

WADC TR 56-454  
ASTIA Document No. AD 110708  
OTS Release

January 1957

SUBJECT: INVESTIGATION OF THE EFFECTS OF HOT-COLD WORK ON THE PROPERTIES OF MOLYBDENUM ALLOYS  
INVESTIGATOR: M Semchyshen  
Robert Q. Barr  
CONTRACT: AF 33(616)-2861  
CONTRACTOR: Climax Molybdenum Company of Michigan  
ABSTRACT: Two alloys, molybdenum-0.28% niobium and molybdenum-0.50% titanium, were investigated from the standpoint of effect of fabrication variables on the degree of strain hardening and the resultant mechanical properties and on subsequent resistance to softening with exposure to elevated temperatures. Fabrication was accomplished either by rolling or forging.

The hardness of the rolled bars increased rapidly with increasing amounts of reduction by rolling up to reductions of about 20%, then increased at a lower rate at successively higher reductions. When rolled bars were subjected to one-hour exposures at elevated temperatures, those sustaining the highest reductions displayed rapid decline in hardness and complete recrystallization in the range from 2250 to 2400°F for the niobium alloy and 2600° to 2800°F for the titanium alloy. Bars sustaining less reduction by rolling generally displayed a gradual approach to minimum hardness with increasing exposure temperature.

Forging studies revealed a rather complex dependence of degree of strain hardening upon composition, specimen condition prior to forging, forging temperature, and amount of reduction. Specimens in the strain hardened condition prior to forging developed substantially higher

hardness than specimens partially or fully recrystallized prior to forging. At the highest forging temperature investigated, 2400°F, specimens from the niobium alloy were less severely strain hardened than comparable specimens from the titanium alloy, owing to the relatively greater structural stability of the latter. A relationship was evolved between as-forged hardness and softening on exposure to elevated temperatures.

Tensile tests at room temperature and 1800°F taken from specimens containing a gradient in the degree of strain hardening invariably failed at locations corresponding to minimum strain hardening. Certain creep-rupture tests at 1800°F, however, failed at locations corresponding to maximum strain hardening. Examination of these specimens indicated that partial recrystallization and softening had occurred under the combined influence of time, temperature and stress at locations where no structural changes had taken place after exposures under similar conditions of time and temperature only.

Throughout the investigation, a consistent tendency was observed whereby the higher the working temperature, the higher the hardness in direct contradiction to classic concepts. This apparently anomalous behavior was attributed to strain aging.

WADC TR 57-40  
ASTIA Document No. AD 118204  
OTS Release

April 1957

SUBJECT: AN INVESTIGATION OF THREE FERRITIC STEELS FOR HIGH-TEMPERATURE APPLICATION

INVESTIGATOR: A. Phillip Coldren  
James W. Freeman

CONTRACT: AF 33(616)-3239

CONTRACTOR: University of Michigan

ABSTRACT: An investigation was carried out to survey the relationships between microstructure and properties at 700° to 1100°F for low-alloy, hardenable steels. A Ni-Cr-Mo (SAE 4340) and two Cr-Mo-V ("17-22-A"S and "17-22-A"V) steels were studied. The results, together with those presented in References 1 and 2 for previous work on 4340 and "17-22-A"S steels, correlate properties with microstructure for three temperatures of isothermal transformation in the pearlite region and three in the bainite region. Oil-quenched and normalized structures were included for comparison. The properties were evaluated for these structures when tempered to both the 300 and 350 Brinell hardness levels. Data for the "17-22-A"V steel in the form of TTT diagram obtained to establish heat-treating condition is included.

These data are supplemented by similar studies for continuous cooling down to rates simulating the air cooling of a 6-inch round. The influence of increasing the heat-treating temperature was also studied. Con-

trolled mixed structures were developed by transforming about 50 percent in the upper bainitic range and about 50 percent in the lower bainitic range. The results of a very limited start for a study of hot-working condition effects is included.

The results are correlated and analyzed in the report. Optimum structures (or treatments) vary with both the alloy and test conditions. In general, bainitic structures of one type or another gave best properties. However, for the Cr-Mo-V steels, pearlites formed at relatively low temperatures were often as strong or stronger than the bainites. Tempered martensite was never the strongest structure and most often was the weakest.

WADC TR 57-58 Pt I  
ASTIA Document No. AD 118289  
OTS Release

May 1957

SUBJECT: NOTCH SENSITIVITY OF AIRCRAFT STRUCTURAL AND ENGINE ALLOYS  
Part I. Preliminary Studies With A-286 and 17-7 PH (TH 1050)  
Alloys  
INVESTIGATOR: Howard R. Voorhees  
James W. Freeman  
CONTRACT: AF 33(616)-3380  
CONTRACTOR: University of Michigan  
ABSTRACT: This program was designed to extend previous analyses of the creep-rupture behavior of notched test specimens held under steady axial load. Experimental studies have also been planned and carried out in an effort to clarify the factors controlling rupture life in the presence of a nonuniform complex stress.

Vacuum melted A-286 alloy produced by the consumable electrode process was chosen for this investigation with the expectation that a range of notch sensitivity could be developed by increasing the temperature of solution treatment. Extensive tests had been planned to study changes in smooth-bar properties corresponding to marked differences in notched-bar strength, in hopes of isolating all the major factors associated with notch sensitivity. For solution temperatures ranging from 1650° to 2300°F, the lot of material studied exhibited strong notch strengthening at 1200°F for nearly all specimens tested with  $K_t$ 's of 1.8, 3.0 and 4.1, despite elongation at fracture as low as 1% or less for certain solution temperature near 2200°F.

Additional smooth- and notched-specimen data were obtained for 17-7PH (TH 1050) sheet material at test temperatures of 600°, 800° and 900°F, and for two small lots of Waspaloy at 1350°F. The 17-7PH alloy exhibited a high degree of freedom from notch sensitivity. The Waspaloy was notched weakened for nominal stresses where yielding occurred at the notch root during load application. For lower nominal stresses, notch strengthening was indicated.

Results obtained indicate that reduction of an alloy's inherent strength by prior plastic deformation may be prominent factor in notch sensitivity. Examination of all available data discloses no case of notch weakening without accompanying loss of life in smooth bars which are prestrained at the test temperature. Conversely, in no known case of marked notch strengthening has the material been found to be weakened by plastic prestrain.

Prestrain effects alone may not be able to explain all notch behavior, but response of the material to plastic strains appears to be a necessary part of any complete analysis of notch effects.

WADC TR 57-154  
ASTIA Document No. AD 118346  
OTS Release

May 1957

SUBJECT: EFFECTS OF PROCESSING VARIABLES ON FRACTURE AND DISC-  
BURSTING CHARACTERISTICS OF FOUR HIGH TEMPERATURE MATERIALS  
INVESTIGATOR: Richard Sakamoto  
CONTRACT: AF 33(616)-2778  
CONTRACTOR: General Electric Department  
ABSTRACT: Generalization of notch sensitivity characteristics of materials evaluated by conventional notch rupture data without due regard to the specific application may represent an unjustified material penalty and unnecessarily preclude the use of a material for a specific application.

Elevated temperature spin test data for several alloys is included which indicate that certain alloys are notch insensitive in thin disc tests even though they show significant strength reduction in the conventional notch rupture tests. This difference in behavior is attributed to the greater constraint existing in the notched cylindrical specimen and indicates that the constraint factor may be fully as significant as the stress concentration.

#### Nonferrous

##### Aluminum

WADC TR 56-99  
OTS Release

June 1956

SUBJECT: MECHANICAL PROPERTY, CORROSION AND WELDING STUDIES ON  
6066 ALUMINUM ALLOY  
INVESTIGATOR: John D. Wood, 1/Lt

**ABSTRACT:** The mechanical properties and stress corrosion characteristics of 6066-T6 extrusion were studied. The effect of salt spray exposure on 6066-T6 sheet was determined and found to approach 2014-T6 alloy. In addition, the welding characteristics of 6066 were investigated and it was found that 6066 alloy can be arc welded using 6066, 716 and 195 alloy filler metals.

WADC TR 56-481  
ASTIA Document No. AD 118192

April 1957

**SUBJECT:** POWDER FABRICATION OF ALUMINUM ALLOYS  
**INVESTIGATOR:** S. G. Roberts  
**CONTRACT:** AF 33(616)-2296  
**CONTRACTOR:** Kaiser Aluminum & Chemical Corp.  
**ABSTRACT:** A new approach to aluminum alloy development, involving the use of powder metallurgy techniques to produce "superalloys" of aluminum, is described; and the preliminary results obtained in an evaluation survey of aluminum alloy systems using this approach are given. These results show that, by this approach, excellent prospects exist for the development of aluminum base alloys having improved mechanical properties at elevated temperatures. The alloy systems found to show promise as a base for alloy development were Al-Mn, Al-Fe, Al-Ti and Al-Cr.

Mixtures of aluminum alloy powders and low density refractory carbides having high moduli of elasticity were fabricated by hot compaction and extrusion, and evaluated by tensile testing at room and elevated temperature (600°F) and by microscopic examination. These fabrications were found to have high moduli of elasticity but were generally inferior in tensile properties to fabrications of the base aluminum alloys without carbide additions.

Extrusions were also prepared from Al-Cu-Mn, Al-Cu-Mg, Al-Mg and Al-Zn-Cu-Mg type alloy powders and evaluated. Significant improvements in the tensile yield strengths at 600°F were found to be effected by powder metallurgy fabrication as compared to conventional wrought fabrication for the Al-Cu-Mn and Al-Cu-Mg type alloys, but no benefits were obtained with the Al-Mg and Al-Zn-Cu-Mg type alloys.

The H<sub>2</sub>O present in the surface film of aluminum alloy powder particles was measured and its effects on the characteristics of subsequent extrusions are discussed.

#### Magnesium

WADC TR 55-207  
ASTIA Document No. AD 118122  
OTS Release

March 1957

SUBJECT: MAGNESIUM ALLOYS WITH HIGH MELTING POINT ADDITIONS  
INVESTIGATOR: R. R. Nash M. C. Huffstutler  
H. K. Adams, Jr. E. J. Tulloch  
A. E. Bibb, Jr.  
CONTRACT: AF 33(616)-2312  
CONTRACTOR: Rensselaer Polytechnic Institute  
ABSTRACT: An exploratory investigation was made to determine the alloying characteristics and the influence on microstructures, mechanical properties and resistance to corrosion of small additions of titanium, hafnium, tantalum, nickel, cobalt, vanadium, boron, chromium, yttrium, rhenium, niobium, tungsten and molybdenum to magnesium and to selected magnesium-base alloys.

A potentially attractive method for the pronounced grain refinement of as-cast structure of magnesium-base alloys was found. Additions of small amounts of titanium dioxide and several forms of vanadium produced a moderate refinement in unalloyed magnesium. These additions were particularly effective when made to magnesium alloys with one or more soluble components. The mechanism of this action was believed to be extensive grain nucleation by stable, foreign nuclei in constitutionally supercooled liquid.

The solubility of titanium, hafnium, vanadium, boron, chromium, niobium, tungsten and molybdenum in magnesium was believed to be extremely limited. Additions of these elements to magnesium and to selected magnesium-base alloys did not influence significantly the mechanical properties or resistance to corrosion of hot rolled sheet. A consistent increase in tensile mechanical properties without loss in ductility in the as-extruded condition was associated with increased chromium additions to AZ31 composition reaching an optimum threshold at approximately 0.3 to 0.5% chromium, intended additions. The retention of rhenium and tantalum in magnesium and magnesium alloys was also believed to be of low magnitude and no improvement in mechanical properties or general resistance to corrosion was observed. Exceptionally high resistance to corrosion was exhibited by two alloys with additions of rhenium and two alloys with additions of tantalum to unalloyed magnesium in the form of hot rolled sheet. The result was considered significant but the specific cause of the benefit was not isolated.

Nickel and cobalt exhibited limited solid solubility in magnesium and magnesium alloys. Nickel additions produced a small improvement in room and elevated temperature mechanical properties of hot rolled sheet but lowered sharply the resistance to corrosion. Cobalt additions were less effective in improving mechanical properties and also lowered resistance to corrosion.

Microscopic examination indicated that the solid solubility of yttrium in magnesium was at least 0.5%. Yttrium additions to unalloyed magnesium produced an increase in tensile yield and ultimate strengths of 36 and 26%, respectively, in the form of hot rolled sheet at room temperature.

November 1956

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
INVESTIGATOR: G. S. Foerster H. Baker  
S. L. Couling R. Johnson

CONTRACT: AF 33(616)-2337

CONTRACTOR: The Dow Chemical Company

ABSTRACT: The work reported under this contract is divided into three sections. Section 1 deals with the development of improved Mg wrought alloys. Two Mg-Mn-Th-Zn-Zr wrought alloys--ZE11 (.6Th-.6Zr) in sheet and ZE31 (1Th-.6Zr) in extrusions--have excellent properties at moderate temperatures, unobtainable in any conventional Mg alloy yet developed. Excellent properties at moderate temperatures can also be obtained by pellet extrusion of high temperature Mg alloys such as HZ32XA. High strength Mg-4Zn-1.5Mn-1.5Mn sheet has been developed for room temperature service but is slightly inferior to ZE41XA, a new experimental sheet alloy.

In Section 2, recent measurements of the thermal and electrical properties of several Mg alloys--AZ31A, HK31XA, HZ32XA, EK30A, EZ33A and HM21XA--are reported. Electrical resistivity varies from  $5.3 \times 10^{-6}$  for HM21XA-W24 to  $9.1 \times 10^{-6}$  ohms/cm<sup>3</sup> for cast AZ31A-F at room temperature and from  $9.2 \times 10^{-6}$  to  $12.9$  ohms/cm<sup>3</sup> for the same alloys at 500F. The temperature coefficient of electrical resistivity is essentially constant at  $0.90 \times 10^{-8}$  ohms/cm<sup>3</sup>/°F. The thermal conductivity of these alloys varies in the reverse order of their electrical resistivity and increases with increasing temperature.

The plastic deformation and preferred orientation of wrought Mg alloys are discussed in Section 3. Mechanical twinning is significant (15-20 volume per cent) in the cold rolling of AZ31A and AZ31B. The attempt to measure indirectly twin volume was only partly successful. Bend tests of Mg-2Al sheet indicate that twin volume decreases with increasing temperature but is still appreciable as high as 815F. The role of heterogeneous deformation or banding in compressive working of Mg alloys is also discussed.

November 1956

SUBJECT: A STUDY OF THE FEASIBILITY OF COATING MAGNESIUM WITH HIGH-PURITY ALUMINUM  
INVESTIGATOR: C. F. Powell  
I. E. Cambell  
CONTRACT: AF 33(616)-3309  
CONTRACTOR: Battelle Memorial Institute

**ABSTRACT:** The feasibility of vapor plating magnesium and magnesium-base alloys with aluminum is evaluated on the basis of information available in the literature on volatile aluminum compounds with the stability range required for vapor-plating "atmospheres".

Pyrolysis of aluminum alkyls and of aluminum hydride and its derivatives is considered of sufficient interest to warrant experimental investigation.

Reduction of aluminum halides with magnesium and disproportionation of aluminum monohalides are considered technically feasible but of lesser interest because the conditions for procuring satisfactory coatings are critical.

WADC TR 56-409  
ASTIA Document No. AD 110571  
OTS Release

November 1956

**SUBJECT:** AUTORADIOGRAPHIC STUDY FOR THE DISTRIBUTION OF THORIUM IN MAGNESIUM ALLOYS

**INVESTIGATOR:** Kurt Wolfsbert, 1/Lt  
George John

**ABSTRACT:** An autoradiographic study of magnesium alloys containing 3% and 13% thorium has been performed. The stripping film method was slightly modified because of the extreme reactivity of magnesium. The results show qualitatively that a large percentage of the thorium is located in or very near to the grain boundaries.

WADC TR 56-453  
ASTIA Document No. AD 118011  
OTS Release

January 1957

**SUBJECT:** FACTORS AFFECTING THE FLUIDITY AND HOT CRACKING OF MAGNESIUM ALLOYS

**INVESTIGATOR:** Howard F. Taylor  
Merton C. Flemings, Jr.

**CONTRACT:** AF 33(616)-2958

**CONTRACTOR:** Massachusetts Institute of Technology

**ABSTRACT:** A vacuum fluidity apparatus was used to determine the fluidities of magnesium alloys. The alloy systems studied were:

1. Magnesium-aluminum binary system from 0 to 20% aluminum.
2. Magnesium-zinc binary system from 0 to 20% zinc.
3. Magnesium-aluminum-zinc ternary system up to 20% alloy content.



4. Magnesium-zinc-zirconium system from 0 to 20% zinc.
5. Magnesium-thorium-zirconium system from 0 to 20% thorium.

Fluidity was determined as a function of temperature for each alloy. Then fluidity at 1400°F was plotted as a function of alloy content. Fluidity at 100°F superheat was plotted for those alloy systems for which the liquidus temperature had been established. Comparison of commercial alloys with the experimental alloys was good. Fluidity curves as a function of alloy content were found to vary as the inverse of computed, non-equilibrium freezing range curves.

A hot tear pattern was adapted for studying the effect of geometry on the hot tearing of magnesium alloys, and for determining the susceptibility of various magnesium alloys to hot tearing; the pattern is stressed in simple tension during solidification. The portion of the test casting susceptible to hot tearing is well fed. Hot tearing in Mg-4% Al alloys was shown to increase with decreasing fillet radius, increasing casting length, increasing section size change, and decreasing gas content.

An experimental apparatus was developed for the measurement of alloy rupture stress and ductility at temperatures above and below the solidus. The apparatus permits stress rupture measurements without first cooling the cast alloy to room temperature.

WADC TN 56-461  
ASTIA Document No. AD 110706

January 1957

SUBJECT: THE DETERMINATION OF CALCIUM IN MAGNESIUM BASE ALLOYS  
INVESTIGATOR: Lois A. Keyser  
Charles D. Houston

ABSTRACT: A gravimetric procedure suitable for the determination of trace amounts of calcium in magnesium-base alloys has been developed in this laboratory.

The sample is dissolved in hydrochloric acid and the magnesium is removed by a double precipitation as the phosphate. The calcium is then precipitated at a pH of six as the oxalate and the washed precipitate is titrated with potassium permanganate.

## Titanium

WADC TR 53-230 Pt 3  
ASTIA Document No. AD 118135  
OTS Release

March 1957

SUBJECT: STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET

INVESTIGATOR: John F. Rudy  
Joseph B. McAndrew  
Harry Schwartzbart

CONTRACT: AF 33(616)-206

CONTRACTOR: Armour Research Foundation

ABSTRACT: This investigation follows a previous investigation which covered the weldability effects of the interstitials, carbon, nitrogen and oxygen, when added individually to several titanium base alloys. Included in these alloys were: 5 Al-Ti, 7 Al-3 Mo-Ti, and 25 V-Ti. For the present study a fourth composition, 6 Al-4 V-Ti, was added to complete the list of base alloys which were investigated as reported herein.

To 5 Al-Ti, an alpha alloy, were introduced combination of C, N<sub>2</sub>, and O<sub>2</sub>, intended to total 0.33% interstitial. Each of the five resulting alloys was divided into 3 parts: the first part was vacuum annealed to reduce the H<sub>2</sub> to less than 50 PPM; the second part was hydrogenated to 100 PPM; and the third part was hydrogenated to 200 PPM. Each of the fifteen alloy heat "parts" was then tested mechanically in the following five conditions: as fabricated to 0.063 inch sheet, as-welded, and after three separate post weld heat treatments. Mechanical testing included tensile and free bend tests, both oriented such that the major loading stresses were transverse to the welding direction. Weldability was determined by these mechanical test results.

A similar program was accomplished with the 6 Al-4 V-Ti  $\alpha+\beta$  alloy. Since background data were missing, eleven 6 Al-4V-Ti plus interstitial alloys were produced. Each was divided into a low and a high H<sub>2</sub> part. The 22 alloy parts were similarly tested in five conditions as above.

The base 7 Al-3 Mo-Ti  $\alpha+\beta$  alloy with only hydrogen added was mechanically tested at 3 hydrogen levels times the five conditions; as fabricated to 0.063 inch sheet, as welded, and after three heat treatments. This same program, with different post weld heat treatments, was applied to the base 25 V-Ti  $\beta$  alloy. The total number of thermal history-interstitial content-alloy conditions tested was over 200.

The results indicated generally that:

The 5 Al-Ti becomes not weld ductile when the  $N_2+O_2$  additions total 0.25%. This was true with both 0.049% C and 0.123% C. Some ductility was indicated in both alloys. However, the 6 inch long welded bend specimens were not able to fold to less than one inch end distance separation without failing. Ductile welds were obtained (<1 inch fold) with  $N_2+O_2$  of 0.16% even with moderately high (0.128%) C. A completely brittle alloy was obtained when  $N_2+O_2$  totaled 0.28%.

The 6 Al-4V-Ti alloy becomes weld embrittled at  $C+N_2+O_2$  as low as 0.26% when  $N_2$  is 0.11%. If the  $N_2$  is held lower (<0.09%), a  $C+N_2+O_2$  of approximately 0.29% can be tolerated. Nitrogen appears to be a much more effective embrittler than  $O_2$ . Oxygen up to 0.21% does not cause complete loss of ductility and an 0.17%  $O_2$  alloy was weldable. An 0.16%  $N_2$  (0.256%  $C_2N_2+O_2$ ), however, was brittle.

WADC TR 54-278 Pt 3  
OTS Release

May 1956

SUBJECT: DEVELOPMENT OF TITANIUM-BASE ALLOYS FOR ELEVATED TEMPERATURE APPLICATION  
INVESTIGATOR: William F. Carew  
Frank A. Crossley  
Donald J. McPherson  
CONTRACT: AF 33(616)-2853  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The principal objective of the work reported herein was a determination of the effects on mechanical properties of complexing the  $\alpha$  and  $\beta$  phases of a promising  $\alpha+\beta$  type alloy, Ti-6Al-3Mo. Tin and zirconium were employed as  $\alpha$  complexers and chromium, manganese, and vanadium were employed as  $\beta$  complexers.

$\alpha$  complexing was found to improve creep resistance and rupture strength, while  $\beta$  complexing reduced these parameters below the levels of the base composition.

In addition, age hardening characteristics of Ti-Al-Ag alloys were determined. Limited creep-rupture data indicated inferiority to a Ti-6Al binary composition.

Further studies on the nature of embrittlement in binary Ti-Al alloys were carried out and results of these studies are reported.

November 1956

SUBJECT: PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS

INVESTIGATOR: A. J. Griest P. D. Frost  
J. R. Doig C. M. Schwartz

CONTRACT: AF 33(616)-445

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: The age-hardening behavior of a series of alpha-beta titanium alloy was investigated. Hardness, tensile, and X-ray diffraction data were used in following the course of the aging behavior of alloys of the systems Ti-Mn, Ti-Mn-Al, Ti-V, Ti-V-Al, and Ti-Fe. The data presented show the effects of solution temperature, aging temperature and alloy composition on the aging reaction. Comparison of the data for the nominal alloys Ti-8 Mn and Ti8Mn-2Al, as well as other data, indicate that the effect of aluminum is to cause the peak strength and hardness to be maintained over longer aging times and to apparently weaken the tendency for omega phase formation.

In a separate phase of the research, a survey was made of possible contributing factors to beta embrittlement (the low ductility characteristic of high-strength alpha-beta alloys after fabrication or heat treatment in the beta field). It was noted that beta-embrittled materials generally have a combination of microstructural features, such as coarse prior beta grain size or acicular alpha precipitates, which might be expected to impair ductility, especially at high strength levels.

October 1956

SUBJECT: HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS  
Part II. Comparison of Various Methods for Hydrogen Analysis

INVESTIGATOR: John W. Seeger, 1/Lt  
Jack A. Winstead, 1/Lt

ABSTRACT: Descriptions of various analytical methods for hydrogen determination in titanium and its alloys are presented, with information on operating procedures and costs.

Laboratories using these methods in general are in disagreement, and vary considerably in analytical reproducibility. Material non-uniformity in hydrogen distribution from position-to-position in a sheet examined in relation to experimental error is found to be a significant factor. A suggested experiment for a single sheet disputed between two laboratories is given as a model by which appropriate experiments can be devised to incorporate the sources for disagreement found in this investigation.

October 1956

SUBJECT: HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS  
Part III. Strain Aging Hydrogen Embrittlement in Alpha-Beta Titanium Alloys

INVESTIGATOR: Harris M. Burte

ABSTRACT: It is now well recognized that hydrogen contamination in alpha-beta titanium alloys can lead to sudden, brittle fracture during the use of these materials. The strain aging embrittlement which causes such fracture has been investigated.

Strain aging hydrogen embrittlement in alpha-beta titanium alloys has its greatest effect on mechanical properties measured at slow strain rates. It can cause low ductility in room temperature tensile tests and premature brittle fracture in room temperature rupture tests. Fracture due to this process tends to be intergranular. Metallographic examination of many hydrogen contaminated alpha-beta alloys shows no evidence for a third phase either before or after fracture. In at least one alloy, however, a third phase was visible after fracture. Both alloy composition and microstructure affect susceptibility to strain aging embrittlement. Increasing test temperature seems to decrease the tendency towards embrittlement, but increases the rate at which embrittlement can occur. A mechanism for strain aging embrittlement is proposed. Other types of embrittlement which may be caused by hydrogen are mentioned.

WADC TR 55-325 Pt II  
ASTIA Document No. AD 97199  
OTS Release

August 1956

SUBJECT: THE EFFECTS OF INTERSTITIAL CONTAMINANTS ON THE NOTCH-TENSILE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS Part II. Alloy Titanium

INVESTIGATOR: E. P. Klier  
N. J. Feola

CONTRACT: AF 33(616)-2281

CONTRACTOR: Syracuse University

ABSTRACT: The notch-tensile properties of alloy titanium contaminated with oxygen, nitrogen and carbon have been determined. In all six alloys have been studied and the effects of the various contaminants on the notch sensitivity have been presented in discussion of the results for each alloy.

Notch sensitivity has been shown to depend on the tensile strength, and becomes potentially severe as the strength exceeds about 150,000 psi. Metallographic structure and alloy content also profoundly affect notch sensitivity in these alloys.

The effects of testing temperature, contamination level, and strain rate on promoting notch sensitivity in the respective alloys are also discussed.

WADC TR 55-454 Pt 2  
ASTIA Document No. AD 97284

September 1956

SUBJECT: A STUDY OF FACTORS AFFECTING THE UNIFORM ELONGATION OF TITANIUM AND TITANIUM ALLOYS  
INVESTIGATOR: A. J. Opinsky  
L. Sama  
L. L. Seigle  
CONTRACT: AF 33(616)-2352  
CONTRACTOR: Sylvania Electric Products, Inc.  
ABSTRACT: In a study of factors which affect the limit of uniform elongation of titanium and titanium-base alloys, iodide titanium is shown to have the largest uniform elongation. Other materials of the high uniform elongation class generally had low strengths, although the metastable  $\beta$ -alloys did exhibit moderate strength. The much lower values of the sponge alloys (outside of the metastable  $\beta$ -alloy) are ascribed to interstitial contents.

The effects of grain size and grain shape are secondary to those of alloying and temperature of test. Strain-induced decomposition of metastable  $\beta$  is shown to be a suitable means of increasing the elongation at maximum load. The other idea of increasing the uniform elongation by strain-induced age hardening is still untested; the apparently most likely system failed to age-harden, or even strain-age harden. In the anisotropy studies, maximum uniform elongation and divergence between yield and ultimate strengths is found in the rolling direction of the sheet.

WADC TR 55-458 Pt II  
ASTIA Document No. AD 110673  
OTS Release

December 1956

SUBJECT: RELAXATION BEHAVIOR OF TITANIUM ALLOYS  
INVESTIGATOR: Franklin J. Gillig  
CONTRACT: AF 33(616)-2340  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.  
ABSTRACT: A relaxation test unit has been designed which provides practically continuous control of the total strain by automatically decreasing the load as the specimen tends to elongate due to creep. The unique feature of this equipment is the ability to apply the initial load in a matter of seconds so that very little relaxation occurs during loading.

Relaxation tests have been made using this equipment for three titanium compositions: A-70, C-130AM, and A-110AT. The A-70 and A-110AT were heat treated to produce two different grain sizes and the C-130AM was heat treated to three different grain sizes. Relaxation tests were made on the three compositions in all of the microstructural conditions at room temperature, 600, and 800°F. The effect of grain size on relaxation characteristics was found to be much the same as in creep testing where the larger grain sizes possess greater resistance to deformation only at the higher test temperatures.

WADC TR 55-510  
ASTIA Document No. AD 97200  
OTS Release

August 1956

SUBJECT: INVESTIGATION OF STRESS RELIEF PROCEDURES FOR TITANIUM AND TITANIUM ALLOYS  
INVESTIGATOR: Franklin J. Gillig  
CONTRACT: AF 33(616)-2688  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.  
ABSTRACT: The causes and effects of residual stresses in titanium aircraft parts were critically examined and a means of reducing these residual stresses to a practical value by thermal treatments was evolved. The purpose of the work was twofold: (a) To present a summary of residual stress effects and attempted residual stress measurements in titanium aircraft parts; (b) To obtain experimental data on the effectiveness of various time-temperature combinations for stress relief of elastically restrained unalloyed and alloyed titanium sheet.

The fundamental principles underlying the build-up of residual stress are reviewed. The difference between macro and micro stresses is discussed and the relationship between these stresses and the properties of the material are pointed out. Methods of relieving residual stresses are outlined. The X-ray diffraction, brittle lacquer and strain relaxation methods of residual stress analysis were attempted. All but the latter were unsuccessful.

The method of setting up simulated residual stresses in a simple beam specimen and measuring their decay with time and temperature is described. Using this method, relaxation tests were made on four titanium compositions: A-55, C-110M, 6 Al-4 V and A-110AT. Three stress levels corresponding to the room temperature elastic limit and approximately 50 and 75% of the room temperature tensile yield strengths were imposed on the test specimens as simulated residual stresses. As a result of these tests, recommended stress relief treatments are given for the above alloys.

June 1956

**SUBJECT:** THE COMBINED EFFECTS OF CARBON, OXYGEN, NITROGEN AND HYDROGEN ON THE PROPERTIES OF TITANIUM SHEET WELDMENTS

**INVESTIGATOR:** John F. Rudy, 1/Lt

**ABSTRACT:** Four series of interstitial alloys were melted and rolled into sheet. The first series contained three levels of hydrogen ranging up to 0.063 weight percent. The second series contained three levels of the elements oxygen and carbon combined, ranging up to 0.200% oxygen and 0.239% carbon. The third series contained oxygen and nitrogen in three levels ranging up to 0.166% oxygen and 0.120% nitrogen. The fourth series contained nitrogen and hydrogen in three levels ranging up to 0.102% nitrogen and 0.088% hydrogen. In addition to these alloys, a base metal ingot which contained no intentional addition was also melted and rolled into sheet.

Inert gas shielded arc welds were made in 1/8 inch sheets of each of the thirteen alloys. The physical properties of these alloys were determined by bend tests, tensile tests, impact tests, hardness measurements and metallographic observations for both the welded and unwelded material. These data will aid in establishing maximum allowable combined percentages of carbon, oxygen, nitrogen, and hydrogen for weldable titanium sheet.

WADC TR 56-146  
OTS Release

June 1956

**SUBJECT:** PROPERTIES OF ACTIVE EUTECTOID TITANIUM ALLOYS

**INVESTIGATOR:** R. F. Bunshah  
H. Margolin

**CONTRACT:** AF 33(616)-2766

**CONTRACTOR:** New York University Research Division

**ABSTRACT:** The microstructure and mechanical properties of Ti-5Cu-3Al, Ti-8Cu-3Al, Ti-5Cu-3Al-2Sn and Ti-8Cu-3Al-2Sn alloys were studied. Room-temperature and elevated-temperature properties are quite comparable to those of annealed commercial alloys. A stable alpha-plus-compound structure shows the best combination of strength and ductility. In order to obtain highest ductility it is necessary to avoid long semi-continuous paths of compound. T-T-T curves and microstructural observations are presented.

Microstructure and mechanical properties of binary Ti-Ni alloys were investigated. They are quite similar to those of binary Ti-Cu alloys.

In line with some recent work on steel, nickel and zinc, some preliminary data on a binary Ti-2Cu alloy show a considerable increase in the strength of this alloy without loss of ductility, by a suitable prestrain and anneal treatment. The strengthening may be attributed to sub-boundaries produced thereby.

WADC TR 53-373 Sup 4



October 1956

SUBJECT: AN INVESTIGATION OF THE USEFULNESS OF ELECTRON MICROSCOPY  
IN THE STUDY OF TITANIUM-ALLOY STRUCTURES

INVESTIGATOR: Frank C. Holden Charles M. Schwartz  
Arthur P. Young Robert I. Jaffee  
Horace R. Ogden

CONTRACT: AF 33(616)-2900

CONTRACTOR: Bettelle Memorial Institute

ABSTRACT: The electron microscope has been found useful in the study of a number of titanium-alloy structures. Those structures that contain fine particles or that otherwise require high magnification and resolution for complete study are candidates for electron microscopy.

Among the subjects investigated, three appeared of particular interest: (1) the decomposition of martensite, (2) the aging process in alpha-beta and beta alloys, and (3) studies of deformation processes in two-phase alloys.

Suitable techniques for electron microscopy already have been developed, but further work is desirable. It seems probable that the electron microscope will take its place among the other useful tools in the research on titanium alloys

January 1957

SUBJECT: THE EFFECT OF MICROSTRUCTURAL VARIABLES AND INTERSTITIAL  
ELEMENTS OF THE FATIGUE BEHAVIOR OF TITANIUM AND COMMERCIAL  
TITANIUM ALLOYS

INVESTIGATOR: Charles B. Dittmar  
G. William Bauer  
Dillon Evers

CONTRACT: AF 33(616)-2922

CONTRACTOR: Mallory-Sharon Titanium Corporation

ABSTRACT: The effect of microstructure on the fatigue behavior of the commercial alloys Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-3Mn Complex has been investigated. Microstructure was found to have little effect on the endurance limit of these alloys unless the structure was extremely coarsened or embrittled.

The effect of interstitial contents representative of the basis for commercial titanium specification, i.e. .07% N<sub>2</sub>, .20% O<sub>2</sub>, .20% C taken singly or grouped together, has been investigated for unalloyed titanium and the commercial alloys Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-3Mn Complex. Titanium and the three titanium alloys investigated generally show increased or unchanged fatigue life at all of the interstitial contents specified above.

August 1956

SUBJECT: STABILITY OF COMMERCIAL ALPHA-BETA TITANIUM ALLOYS  
INVESTIGATOR: Donald A. Wruck, 1/Lt.  
ABSTRACT: An investigation was undertaken to observe the influence of various factors such as temperature, time-at-temperature, stress, and hydrogen contamination on the stability characteristics of commercial alpha-beta titanium alloys, as well as to gain an insight into the nature of the reactions responsible for the ductility loss in an unstable microstructure. Commercial alloys utilized in one phase or another in this program included Ti-150A, Ti-155A, Ti-140A, C-130AM, C-110M, Ti-6Al-4V, RS-140X, and 3Mn Complex. Tests show that in the absence of hydrogen contamination the alpha-beta type alloys can be rendered quite stable up to the maximum temperatures at which they retain useful strength for stressed applications. The Ti-150A alloy loses a considerable amount of room temperature ductility after being exposed to 800°F for 200 hours and can subsequently be rendered ductile by annealing at 1200°F for 24 hours. Mechanisms of embrittlement are discussed, as well as the practical significance of stability.

WADC TR 56-545  
ASTIA Document No. AD 110737  
OTS Release

January 1957

SUBJECT: DEVELOPMENT OF A HEAT-TREATABLE TITANIUM ALLOY HAVING ADE-  
QUATE FORMABILITY  
INVESTIGATOR: Herbert A. Robinson Alvin M. Sabroff  
Andrew J. Griest Paul D. Frost  
CONTRACT: AF 33(616)-2901  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: A series of 27 experimental alpha-beta titanium alloys were melted, forged, rolled to sheet, and evaluated as potential formable and heat-treatable sheet alloys. Formability was evaluated by true stress-true strain tensile data and by bend tests. In general, the solution temperature had greater effect than alloy composition on the relative formability of the alloys in the as-quenched condition. However, several compositions appeared to offer greater promise than the others from the standpoints of formability and ability to be aged, after forming, to a high strength level. A Ti-3Fe-3Mo-2Al alloy was of particular interest. Additional research on this and other alloys is recommended.

WADC TR 56-580  
ASTIA Document No. AD 118118  
OTS Release

March 1957

SUBJECT: THE EFFECT OF VARIOUS HEAT TREATMENT CYCLES UPON THE MECHANICAL PROPERTIES OF TITANIUM ALLOYS WITH VARIOUS INTERSTITIAL LEVELS

INVESTIGATOR: Benjamin F. Hadley  
G. William Bauer  
Dillon Evers

CONTRACT: AF 33(616)-2795

CONTRACTOR: Mallory-Sharon Titanium Corporation

ABSTRACT: The effect of various heat treatments on the mechanical properties of 5/8" diameter bar from four titanium alloys of five interstitial content levels each was determined. Three major types of heat treatment were employed: (1) a solution treatment and age cycle, (2) a step quench cycle and (3) a solution treatment, isothermal transformation cycle. The alloys investigated were Ti-5Al Complex (Ti-5Al-1.7Cr-1.6Fe-1.6Mo), Ti-3Mn Complex (Ti-3Mn-1Cr-1Fe-1Mo-1V), Ti-6Al-4V and Ti-4Al-4Mn. Interstitial levels were (1) low (100 EHN sponge base), (2) high oxygen (.24%), (3) high nitrogen (.07%), (4) intermediate oxygen and nitrogen (.16% and .04% respectively) and (5) high carbon (.2%).

Three hundred hour creep tests were performed on materials of interstitial levels 1, 2, and 4. The elevated temperature tensile properties, room temperature notch strength, and Charpy impact data of material of interstitial level 4 were determined. Beta transi, T-T-T curves and end quench curves were determined for all material.

The effect of section size on the mechanical properties and the heat treatment response of the Ti-5Al Complex alloy was determined.

WADC TR 56-597  
ASTIA Document No. AD 110748  
OTS Release

February 1957

SUBJECT: STUDIES OF FACTORS AFFECTING THERMAL STABILITY OF TITANIUM-BASE ALLOYS

INVESTIGATOR: Frank C. Holden  
Horace R. Ogden  
Robert I. Jaffee

CONTRACT: AF 33(616)-3208

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Studies were made to determine the factors that influence the stabilization and the thermal stability of alpha-beta titanium alloys. Thermal stability for three commercial alloys MST-6Al-4V, Ti-155A, and C-130AM, was good for three heat-treatment conditions under creep exposure tests through 800 F. Some loss of ductility was observed for the Ti-155A and C-130AM alloys fabricated in the beta field and annealed in the alpha-beta field.

Substitutions of various beta stabilizers in a Ti-5Cr-5Mo alloy were made to check their effects on thermal stability. The active eutectoid formers, nickel, copper, and chromium, were detrimental; thermal stability was improved by substitutions of manganese or molybdenum, and by aluminum additions.

Small amounts of strain, introduced either before equilibration or before stabilization, had no significant effect on thermal stability of a Ti-5Cr-5Mo alloy.

#### Vanadium

### PACKAGING

WADC TR 53-133 Pt 3  
ASTIA Document No. AD 97334  
OTS Release

September 1956

SUBJECT: INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES Part 3 Investigation of the Effects of Molecular Weight, Chain Branching, and Irradiation on Polyethylene With Regard to Shelf Life in Bottles

INVESTIGATOR: Jules Pinsky  
A. R. Nielsen  
J. H. Parlman

CONTRACT: AF 33(616)-112

CONTRACTOR: Plax Corporation

ABSTRACT: Part I of this contract was concerned with the Shelf Life of Liquids and Solids in blown Plax 4-ounce bottles made from Bakelite's DE-2400. For this investigation, Part 3, the effects of higher molecular weight (Bakelite's DE-2450), side chain branching (Alathon 10, Alathon S-1439, and Alathon S-1447), irradiation by electron bombardment (High Voltage Irradiated DE-2450) were studied. The changes in permeability factors occasioned by the above variations are presented and are tabulated in comparison with the previous results on Bakelite's DE-2400. It can be seen from this report that, while there are considerable differences in permeability, the packageability status is not generally changed.

September 1956

SUBJECT: INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES Part 3, Sup 1. Theoretical Investigation of the Effects of Molecular Weight, Side Chain Branching, and Irradiation on the Mechanism of Transfer of Materials Through Polyethylene

INVESTIGATOR: Dr. Henry A. Bent  
Jules Pinsky

CONTRACT: AF 33(616)-112

CONTRACTOR: University of Connecticut  
Plax Corporation

ABSTRACT: In this study P-Factors and absorption tests reported in Part 3 are analyzed and discussed, together with some data on aqueous solutions from Part 1. The theory of mass transfer through polyethylene is extended, particularly with regard to the  $\log P_o$  vs  $E_p$  plot, and the results applied to recent data on carbon tetrachloride.

It is found that chain branching and irradiation increase swelling coefficients and diffusion constants at low temperatures, but decrease them at very high temperatures. The importance of placing room temperature P-Factors in this perspective is stressed. It is suggested that activated diffusion through polyethylene induces structural changes in the polymer not unlike local melting. Thermodynamical reasoning also indicates that frequency and P-Factors increase with increasing rigidity and mass of penetrant. In both respects carbon tetrachloride shows this increase with polyethylene.

WADC TN 55-216  
ASTIA Document No. AD 97252

September 1956

SUBJECT: THE PERFORMANCE OF STRAPPED, NAILED, WOODEN BOXES HAVING SIDES, TOPS, AND BOTTOMS OF 7/16- OR 9/16-INCH RESAWN LUMBER

INVESTIGATOR: R. K. Stern  
D. O. Yandle

CONTRACT: PQ(33-600)-53-4023

CONTRACTOR: Forest Products Laboratory

ABSTRACT: The strength of 7/16- and 9/16-inch resawn lumber when used as box sides, tops, and bottoms was compared by rough-handling tests of 112 strapped, nailed, wooden boxes.

Boxes made of 9/16-inch lumber were much more resistant to rough-handling than boxes made of 7/16-inch lumber.

January 1957

SUBJECT: MAGNETIC SHIELDING OF MAGNETRON TUBES AND MAGNETS FOR SHIPMENT BY AIR  
INVESTIGATOR: A. W. Voss  
CONTRACT: PO(33-600)53-4023  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: A method is presented for shielding magnets and magnetron tubes containing magnets to protect sensitive aircraft instruments from being affected by stray fields of the magnets. A survey showed that current military practice is to stow magnets at least 7 feet from the compass-sensing device. To meet the criterion established by WADC, the stray fields have to be limited to not more than 5.25 milligauss at a distance of 7 feet from the magnets. Various methods and materials were tested, and it was found that shields of low-carbon steel would confine the stray fields and prevent adverse effects on aircraft instruments. A method of forming these shields is described and illustrated. The minimum weight and number of layers required for various tubes and magnets is tabulated, and methods are presented by which the shielding required for similar tubes and magnets can be established.

WADC TN 55-706  
ASTIA Document No. AD 110525

November 1956

SUBJECT: EVALUATION OF FOUR DESIGNS OF EXTERIOR SHIPPING CONTAINER FOR HUMAN REMAINS TRANSFER CASE  
INVESTIGATOR: T. B. Heebink  
CONTRACT: PO(33-600)-53-4023  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Four boxes, each of different design, were evaluated as exterior containers for the human remains transfer case. The evaluation tests consisted of a high-moisture-content conditioning test and the following rough-handling tests: superimposed load; handle; flat, edgewise- and cornerwise-drop; and pendulum impact.

Three of the boxes swelled objectionably when the moisture content was increased from 7-1/2 to 19 percent. The same three boxes showed either racking distortion or residual deflection in the rough-handling tests. The fourth box, of cleated plywood construction, was superior in every way. It showed very little weakness in the moisture test or the rough-handling tests, was 19 percent lighter, and is possibly cheaper to build.

WADC TR 56-342  
ASTIA Document No. AD 97327  
OTS Release

September 1956

SUBJECT: THE THEORY AND OPERATION OF A DYNAMIC TESTER FOR EVALUATING  
PACKAGE CUSHIONING MATERIAL  
INVESTIGATOR: A. D. Klingenberg, 1/Lt  
ABSTRACT: The principles of cushioning design are presented. The characteristics of several possible types of dynamic cushion testers are discussed, and the reasons shown for selection of the free fall platform tester. A description of the tester and the dynamic cushion test procedure is given. A method for data reduction of test results is shown. The reliability of the cushion tester is proven by statistical techniques. Sample drop test results are given. It is shown how deflections caused by dropping a series of weights can be computed on the basis of a measured deflection occurring in the first drop.

WADC TR 56-357  
ASTIA Document No. AD 118234  
OTS Release

April 1957

SUBJECT: EVALUATION OF AUTO-NAIL FOR FABRICATING THE PANELS OF CLEATED  
PAPER-OVERLAID-VENEER BOXES  
INVESTIGATOR: Alvin A. Mohaupt  
CONTRACT: PO(33-600)-53-4023  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Tests were conducted to determine the suitability of Auto-Nails, as compared to regular nails, for fastening cleats to paper-overlaid veneer for use as box panels. Eight paper-overlaid-veneers were included in lateral-displacement tests from which it was found that approximately three Auto-Nails equaled the lateral resistance afforded by two regular nails. Drop tests of cleated, paper-overlaid-veneer boxes with panels made of Auto-Nails with 2- and 3-inch spacings showed that these boxes performed as well as or better than cleated-plywood boxes fabricated with regular nails spaced 3 inches apart.

WADC TR 56-464  
ASTIA Document No. AD 110586  
OTS Release

November 1956

SUBJECT: MEASURING FIELD HANDLING AND TRANSPORTATION CONDITIONS  
INVESTIGATOR: Donald S. Martin, 1/Lt  
ABSTRACT: The proof that an item is properly packaged for shipment is its ability to withstand a drop test. The height from which drop tests are conducted is arbitrarily chosen and may overstate the facts. A more realistic height of drop could be chosen if recording instruments were concealed inside ordinary shipping containers and shipped, where the instrument would record the impacts due to rough handling of packages by handling personnel.

WADC TR 53-373 Sup 4

The bottleneck to an instrumented shipping program is instrumentation. Reported, herein, is the development of an instrument of sufficient accuracy to measure rough handling in terms of inches of drop height. Cost of the instrument is \$200 each.

The statistical shipping plan for using the instrument and a method of relating the data obtained to container design is also discussed.

WADC TR 56-647  
ASTIA Document No. AD 118345  
OTS Release

May 1957

SUBJECT: EVALUATION OF FIVE TYPES OF CONTAINERS FOR JETTISONABLE  
FIBERGLASS FUEL TANKS

INVESTIGATOR: T. B. Heebink

CONTRACT: PO(33-600)-53-4023

CONTRACTOR: Forest Products Laboratory

ABSTRACT: Five types of containers for jettisonable fiberglass fuel tanks were evaluated and compared to determine whether savings could be made in tare weight and cubic displacement over the container now in use for aluminum tanks. Present requirements are for a Specification MIL-C-4349 type of crate. Each of the containers was tested for resistance to rough handling by the edgewise drop, cornerwise drop, and pendulum impact tests. One type of open crate and a cleated plywood box performed best under rough handling. The cleated plywood box had the least tare weight and cube. The open crate would be satisfactory, and could be used if it is found necessary to have unlimited ventilation in storage to minimize corrosion of metal fittings.

WADC TR 57-51  
ASTIA Document No. 118166  
OTS Release

March 1957

SUBJECT: EVALUATION OF THE TWIST LOCK CONTAINER

INVESTIGATOR: Warren D. Hypes

ABSTRACT: The twist-lock type rigid metal container qualifying as a Method IIId unit protection in accordance with Specification MIL-P-116 was evaluated. The Forest Products Laboratory conducted a field service test, the results of which indicate acceptance of the twist-lock container by the majority of depot personnel. The field service test also concluded that the quality of the gasket, in conjunction with the closing technique, was the most important factor in obtaining a satisfactory seal. The Laboratory tests, conducted by the Forest Products Laboratory and Wright Air Development Center, concluded that the twist-lock container will meet the performance requirements for Method IIId unit protection.



## PAINT REMOVER

## PETROLEUM PRODUCTS

### Antisizeze Compounds

WADC TR 55-493  
ASTIA Document No. AD 97213

August 1956

SUBJECT: EVALUATION OF COMPOUNDS FOR USE IN HIGH TEMPERATURE ANTI-  
SEIZE APPLICATIONS

INVESTIGATOR: Gary G. Winters, 2/Lt  
Edward Ogletree  
R. C. Zurbrigg

ABSTRACT: A laboratory mechanical evaluation test method has been developed by this Center for evaluating anti-seize compounds for use in high temperature applications such as threaded fasteners. Data obtained from a typical gas turbine engine application have established that the developed test method will qualitatively predict the suitability of anti-seize compounds for use at temperatures up to 1500°F. On the basis of the results of this progress, several commercially available anti-seize compounds are recommended for use at high temperature. A recommendation is also made that a performance specification be written for high temperature anti-seize compounds incorporating the developed mechanical evaluation test as a requirement.

The purpose of this report is to evaluate the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

### Fuels

WADC TR 53-63 Pt 3  
ASTIA Document No. AD 118084

February 1957

SUBJECT: STABILITY OF JET TURBINE FUELS

INVESTIGATOR: Harry B. Minor  
Alan C. Nixon  
Roy E. Thorpe

WADC TR 53-373 Sup 4

CONTRACT: AF 33(616)-2707  
CONTRACTOR: Shell Development Company  
ABSTRACT: The Emeryville Research Center of Shell Development Company has been investigating the effect of hydrocarbon type, minor constituents, additives and treatment on the storage and high temperature stability of jet turbine fuels.

A heat exchanger test developed here has been used for high temperature stability testing. Although most pure hydrocarbons are more stable in this test than commercial fuels,  $\alpha$ -methyl naphthalene is equally unstable. Small amounts of chromatographic polar fractions reduced the high temperature stability of a stable straight-run fraction; olefin and olefin-diolefin fractions were inert. Dispersants are effective in prolonging filter plugging time although the rate of deposit accumulation on the filter is not changed. Silica gel treatment had a beneficial effect on high temperature performance, but not sodium treatment.

The one successful storage inhibitor was again found effective under mild conditions of aging. Metal deactivator is effective in preventing catalytic action by copper-containing steels but is only partially effective against copper surfaces. The deactivator increases the rate of solution of copper by a factor of about five. With dissolved copper, at least 2.0 times the stoichiometric amount of deactivator is required. The final results of the five year desert storage program are discussed briefly with respect to the effect of fuel type and source, oxygen availability and filterability.

Chromatographic separation of catalytically cracked, thermally cracked and straight run gas oils into type components shows that conjugated diolefins, aromatic olefins and "resin fractions", consisting largely of nitrogen bases and phenols, are responsible for instability and lack of anti-oxidant susceptibility under mild aging conditions. Objectionable olefinic constituents are not appreciable in straight run gas oils. They are more prevalent in thermally cracked than in catalytically cracked gas oils.

WADC TR 53-63 Pt IV  
ASTIA Document No. AD 118085  
OTS Release

February 1957

SUBJECT: STABILITY OF JET TURBINE FUELS Part IV Thermal and  
Oxidative Stability  
INVESTIGATOR: Harry B. Minor  
Alan C. Nixon  
Roy E. Thorpe  
CONTRACT: AF 33(616)-2707  
CONTRACTOR: Shell Development Company

**ABSTRACT:** The Emeryville Research Center of Shell Development Company has been investigating the effects of hydrocarbon types, minor constituents, additives and treatment on the storage and high temperature stability of jet turbine fuels.

Previously it was shown that conjugated diolefins, conjugated aromatic olefins, alkyl thiophenes, and "resin" fractions were responsible for instability and lack of antioxidant susceptibility under mild aging conditions. To provide a better understanding of the adverse nature of the "resins", this fraction has been separated into its basic and acidic components and a neutral fraction consisting in part of an unknown polymeric heterocyclic fraction and partly of aromatic hydrocarbons and thiophenes, similar to the main hydrocarbon fraction. The nitrogen bases were resolved into aniline homologs, and pyridines-quinolines. The nitrogen bases do not affect stability adversely; the natural phenols have a small effect only; but the heterocyclic neutral "resin" fraction is extremely unstable.

The nitrogen picture was completed by isolating and evaluating the pyrroles which, together with the bases, comprise the bulk of the nitrogen compounds. Pyrroles are markedly unstable, forming large amounts of insolubles.

With respect to the high temperature performance of the above components, aromatic olefins in combination with aromatics, phenols, nitrogen bases and pyrroles decrease the stability of an aromatic-free solvent. An anilines-rich fraction had a moderate adverse effect whereas pyridines-quinolines appeared to exert a pronounced stabilizing action. Prior aging of jet fuels causes a large decrease in thermal stability which cannot be anticipated from the small increases in gum and peroxide content. Of several additives tested only one offered some benefit. A number of other additives are being examined to offset the effect of storage; a few compounds showed some immediate beneficial action with respect to heat exchanger fouling. The effective keto-imine storage stabilizer had an immediate adverse effect on the thermal stability of 3 of 6 CRC fuels tested although it reduced gum formation in the same 3 fuels (and one other) during storage. In the one case where this compound improved high temperature performance somewhat, there was an adverse effect on the formation of insoluble gum during storage. In other fuels the keto-imine generally has no effect on high temperature stability although it may significantly reduce formation of gum on aging.

A few compounds in addition to the keto-imine and its analogs have shown some favorable effect in reducing gum formation in fuels. The beneficial action of the keto-imine may be related to its ability to deactivate soluble copper. The latter has been found in most jet fuels in amounts ranging from about 0.05 to 0.25 ppm. The conventional commercial metal deactivator has not always been effective in reducing gum formation.

Repeated exposure (recycle) of jet fuels to high temperature conditions (ca 450°F) is more severe than once-through from the standpoint of rate of deposition on both filters and preheaters, but once-through is generally more severe on the rate of filter plugging ( $\Delta$  P).

WADC TR 53-63 Pt V  
ASTIA Document No. AD 118230

April 1957

SUBJECT: STABILITY OF JET TURBINE FUELS Part V. Effect of Nuclear Radiation  
INVESTIGATOR: Alan C. Nixon  
Roy E. Thorpe  
CONTRACT: AF 33(616)-2707  
CONTRACTOR: Shell Development Company  
ABSTRACT: The Emeryville Research Center of Shell Development Company is studying the effects of ionizing radiation on jet fuels and their components, particularly with respect to radiation-induced changes in thermal stability. This investigation is progressing in three directions:

1. Evaluation of radiation damage in terms of thermal and oxidative stability.
2. Determination of the chemical changes by spectroscopy and physical separations.
3. Evaluation of the feasibility of "radiation protectors" (e.g., aromatic compounds).

Differences are noted between the damage suffered by various jet fuels, both with regard to chemical changes and stability effects. On the basis of the infrared spectra of the irradiated fuels, three general radiation reactions occur in jet fuels:

1. Formation of carbonyl compounds (in air)
2. Formation of olefins (in both air and nitrogen).
3. Destruction of benzene and naphthalene (particularly in nitrogen).

Irradiation of jet fuels to  $1 \times 10^8$  roentgens increases soluble gum (as measured by chromatogum, with and without prior aging) to about the same level in either air or nitrogen. The insoluble gum level is not altered appreciably in either case.

Of three typical jet fuels, two (a special kerosene and a JP-5 fuel - Los Angeles Basin crude) improved in thermal stability at 450°F as a result of irradiation in air to 10<sup>8</sup> roentgens. A JP-4 fuel - San Joaquin crude - on the other hand, was affected adversely, as judged by filter-plugging tendencies. Filter-plugging time was reduced by a factor of 2 to 3. A start was made towards relating the thermal stability of these fuels to composition changes.

The results of initial studies of "radiation protectors" indicate that the aromatic compounds investigated thus far do not generally extend sponge-type protection to jet fuels; instead they themselves react in substantial amounts on irradiating, in nitrogen or air, jet fuels containing 2.5 to 20%w of added aromatic compounds.

At the present time the best choice for a radiation-stable jet fuel would appear to be one similar to the special kerosene, RAF-99, referred to above.

### Hydraulic Fluids

WADC TR 54-532 Pt III

April 1957

ASTIA Document No. AD 118179

SUBJECT: HIGH-TEMPERATURE HYDRAULIC FLUIDS Part III. Development  
Of Aromatic Ethers as Base Stock

INVESTIGATOR: Edward S. Blake William C. Hammann  
James W. Edwards Thomas Reichard

CONTRACT: AF 33(616)-2623

CONTRACTOR: Monsanto Chemical Company

ABSTRACT: Over fifty alkyl-substituted and unsubstituted aromatic ethers have been synthesized, many of which are new compounds. The melting point, pour point, vapor pressure, viscosity, and thermal stability of most of these ethers have been measured from which correlations have been made by which the boiling point, 210°F viscosity and 210-400°F ASTM slope, and thermal stability of a new ether can be predicted. From the data at hand it should be possible to synthesize aromatic ethers stable to 700°F, liquid at 0°F, viscosity < 550 cs at 32°F and with a vapor pressure < 600 mm/700°F.

WADC TR 55-89 Pt III

July 1957

ASTIA Document No. AD 97101

SUBJECT: PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL  
SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS

WADC TR 53-373 Sup 4

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INVESTIGATOR: H. M. Schiefer, 1/Lt

ABSTRACT: A physical and chemical laboratory evaluation has been conducted by the Materials Laboratory on an experimental high temperature hydraulic fluid, MLO 54-645, blended at Wright Air Development Center in such a manner to pass the MIL-H-8446 (USAF) Specification rubber swell test. This silicate-diester base fluid was evaluated primarily against the requirements outlined in the above specification for a -65° to 400°F (200-500 hours) non petroleum base hydraulic fluid. This fluid passes all specification tests with the exception of the oxidation test at 400°F in which the fluid gave a marginal neutralization number increase and viscosity decrease.

Some additional tests were performed on this fluid, which indicated that, as for other silicate fluids tested, the hydrolytic stability at 400°F seems to be one of the major deficiencies of this type of fluid. It is apparent from mock-up and 400°F laboratory hydrolytic stability tests, that the present hydrolytic stability test at 200°F needs revision. Some wear data were obtained with this fluid at 167° and 400°F which indicated that the fluid should have sufficient lubricity to lubricate high temperature pumps. Some minimum spontaneous ignition temperatures obtained with the fluid when sprayed from varying pressure levels into a glass vessel at atmospheric pressure, showed that the ignition temperature of this fluid decreases to approximately 510°F at high pressures. This phenomenon is also present in other silicate base fluids tested under these conditions.

WADC TR 55-89 Pt IV  
ASTIA Document No. AD 118123  
OTS Release

March 1957

SUBJECT: PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL  
SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS Part IV  
MLO 8200

INVESTIGATOR: H. M. Schiefer, 1/Lt  
R. J. Benzing

ABSTRACT: A physical and chemical evaluation has been conducted by the Materials Laboratory on an experimental high temperature hydraulic fluid, MLO 8200, which has a disiloxane base fluid. It was evaluated against the requirements of Specification MIL-H-8446 (USAF) and for higher temperature applications to 550°F. It passes all the requirements of MIL-H-8446 with the exception of rubber swell.

Some additional tests were performed on this fluid which indicated that, as for other silicate fluids tested, the hydrolytic stability at 400°F seems to be one of the major deficiencies of this type of fluid. It is apparent from mock-up and 400°F laboratory hydrolytic stability tests that the present hydrolytic stability test at 200°F needs revision. Some wear data were obtained with this fluid at 167° and 400°F which indicated that the fluid should

have sufficient lubricity to lubricate high temperature pumps. Some minimum spontaneous ignition temperatures obtained with the fluid when sprayed from varying pressure levels into a glass vessel at atmospheric pressure showed that the ignition temperature of this fluid decreases to approximately 510°F at high pressures. This phenomenon is also present in other silicate base fluids tested under these conditions.

WADC TR 56-25  
OTS Release

May 1956

SUBJECT: EVALUATION OF CHLOROPHENYL PHOSPHATES AS POTENTIAL BASE STOCKS FOR HIGH TEMPERATURE HYDRAULIC FLUIDS

INVESTIGATOR: Dr. J. C. Dacons  
H. M. Schiefer, 1/Lt

ABSTRACT: Three phosphate ester fluids, all containing phenyl and/or chlorophenyl groups have been evaluated for potential high temperature hydraulic fluid application. These fluids are so chosen that they represent three different classes of chlorophenyl phosphate fluids and results obtained form a basis for predicting the success or failure of the chlorophenyl phosphates in general for this application. The fluids were tested extensively for thermal and oxidative stability since these are the areas in which they were particularly controversial. Other tests include viscosity, pour point, flash point, fire point, and foaming tendency determinations. Results indicate good thermal stability in glass apparatus only and poor thermal and oxidation stability particularly at high temperatures in the presence of metals. The flash points, fire points, and foaming tendencies are good, but the viscosity-temperature relationships are poor. Some wear data were obtained but are inconclusive due to the non-availability of equipment for testing at high temperatures. Such equipment is in the process of being developed. Low temperature wear test results indicate that these fluids would probably be poor in lubricity at high temperatures, but may have possibilities as extreme pressure additives.

WADC TR 56-168  
ASTIA Document No. AD 118045

February 1957

SUBJECT: SILICONE FLUID RESEARCH FOR THE DEVELOPMENT OF HIGH TEMPERATURE HYDRAULIC FLUID AND ENGINE OILS

INVESTIGATOR: Edgar D. Brown  
Norman G. Holdstock  
John M. McGuire

CONTRACT: AF 33(616)-2892

CONTRACTOR: General Electric Company

ABSTRACT: The evaluation of chlorophenyl siloxane systems showed that this class of silicones showed some improvement in lubricity over previously evaluated dimethyl silicones and phenyl methyl silicones of comparable

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viscosity-temperature characteristics and offered promise in the development of 700°F hydraulic fluids and high bulk oil temperature turbine engine oils. However, improvements were required in thermal stability, oxidation stability, lubricity, and foaming tendencies. Development effort based on improvements of the chlorophenyl silicone Versilube F-50 (No. 81406) by additives has shown that: (a) Reaction of F-50 with tin compounds, such as stannic chloride, yields a product with greatly improved lubricity at 300° to 700°F temperature range over that of the original silicone. However, viscosity-temperature properties are much poorer; (b) Addition of iron octoate improves the oxidation resistance of F-50 at 500°F to within the contract requirements for turbine oil development; (c) Perfluoro acids and amines greatly reduce the foaming tendency. However, these materials are too volatile within the desired temperature limits for turbine oil use; and, (d) F-50 type chlorophenyl silicones become thermally unstable and undergo rearrangement and degradation in the 600°-620°F range. None of the materials studied showed any improvement in thermal stability over the initial starting material.

These additive investigations indicate that a -30° to 600°F hydraulic fluid can be prepared from a suitably modified chlorophenyl silicone of Versilube F-50 structure. The basic compound, F-50, has -65° to 550°F potential. Further development of chlorophenyl silicone formulations will be aimed at improving this performance.

WADC TR 56-207

October 1956

ASTIA Document No. AD 110420

SUBJECT: DEVELOPMENT OF "CHAIN TYPE" POLYPHENYL COMPOUNDS FOR USE AS HIGH TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS

INVESTIGATOR: Josef J. E. Schmidt  
John A. Krimmel  
John R. Hobaugh

CONTRACT: AF 33(616)-2939

CONTRACTOR: Denver Research Institute

ABSTRACT: A comprehensive literature survey on biphenyl and other "chain type" polyphenyl compounds was conducted and abstracted data stored in a central card file and on IBM punch cards. Based on the collected data and correlations thereof a series of biphenyl and terphenyl compounds were synthesized and tested for thermal stability and other physical properties.

Alkyl derivatives of biphenyl and of m-terphenyl showed considerable thermal stability. It is suggested that, among the other alkyl-m-terphenyl derivatives, compounds with good thermal stability and wide liquid range may be expected.



May 1957

SUBJECT: DEVELOPMENT OF "CHAIN TYPE" POLYPHENYL COMPOUNDS FOR USE AS HIGH TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS

INVESTIGATOR: Josef J. E. Schmidt  
John A. Krimmel

CONTRACT: AF 33(616)-2939

CONTRACTOR: Denver Research Institute

ABSTRACT: This report describes the synthesis of alkyl biphenyl and terphenyl derivatives and their intermediates. These compounds have been synthesized in the course of investigations for the development of thermally-stable fluids. Modifications for the synthesis of isopropyl-m-terphenyl have been introduced for the achievement of higher yields.

In the terphenyl series the polyalkylation of m-terphenyl has been investigated and isomerization of m-terphenyls attempted.

Thermal stability tests on isopropyl-m-terphenyl and amyl-isopropyl-m-terphenyl have been carried out by heating over a longer period of time at elevated temperatures. These tests indicated that among m-terphenyls substituted in the 4 position, the isopropyl derivative is more stable than the corresponding amyl derivative.

WADC TR 56-634  
ASTIA Document No. AD 118073

February 1957

SUBJECT: HYDRAULIC FLUIDS FOR 400°F. TEMPERATURE SYSTEMS

INVESTIGATOR: H. M. Schiefer, 1/Lt  
Bernard Rubin

ABSTRACT: The properties and some of the performance characteristics of a group of silicate-ester compositions evaluated as -65° to 400°F. hydraulic fluids are described.

The silicate-ester fluids have good viscosity-temperature characteristics, low volatility, and adequate thermal stability for long term use at 400°F.; pour points and -65°F. viscosity are within range for -65°F. use. However, these formulations are hydrolytically unstable in the presence of free water, and show lower lubricity in the 300° to 400°F. range than the presently used mineral oil fluid (Specification MIL-O-5606) has in the 200°F. to 250°F. range. However, mock-up hydraulic systems, adequately engineered and designed, and hydraulic pumps have operated for extended periods at temperatures of 400°F.

Specification MIL-H-8446 has been prepared and issued. Producers of silicate-ester fluids have been requested to submit samples for qualification.

Effects of the silicate-ester fluids on elastomers, protective coatings, fabrics, and plastics are reported to aid in engineering selection of fluid resistant materials.

WADC TR 57-151  
ASTIA Document No. AD 130764  
OTS Release

May 1957

SUBJECT: RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT  
HYDRAULIC FLUIDS  
INVESTIGATOR: Michael G. Zabetakis  
Aldo L. Furno  
Joseph J. Miller, Jr.  
CONTRACT: AF 18(600)-151  
CONTRACTOR: Bureau of Mines  
ABSTRACT: The results of minimum spontaneous ignition temperature tests conducted on seven hydraulic fluids while in contact with seven surfaces found in aircraft under conditions likely to be encountered in practice are presented here. These tests were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 1 November 1955 and 31 October 1956. Hydraulic fluids Esso Univis J-43, conforming to specification MIL-O-5606, MLO 53-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS 45), MLO 54-581, MLO 54-645 (85% Oronite 8200 + 15% Plexol), MLO 54-856 (Hollingshead 72073C), and MLO 8200 (Oronite 8200) were tested while in contact with heated aluminum, beryllium-copper, copper, magnesium, pyrex glass, stainless steel and titanium surfaces. The effects of both test chamber pressure and injection pressure variations were investigated.

### Lubricants

WADC TR 53-83 Pt 4  
ASTIA Document No. AD 97313  
OTS Release

September 1956

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES  
INVESTIGATOR: Edward A. Swakon  
CONTRACT: AF 33(038)-23687  
CONTRACTOR: Standard Oil Company (Indiana)

**ABSTRACT:** In the work directed toward the development of an aircraft grease suitable for use over the temperature range from -65° to 450°F and higher, emphasis was placed on the development and evaluation of arylurea greases made with the best available fluids and the development of thickeners for use as high as 700°F. The chief criteria of laboratory evaluation were performance in the ABEC-NLGI Bearing Tester, the Navy Gear-Wear Tester, low-temperature torque test, apparent viscosity and linear screw-and-nut actuator.

The grease selected to replace MLG-9305 consists of DC 550 Silicone Fluid thickened with para-tolyl and para-chlorophenylurea derivatives of bitolylenediisocyanate. It was designated MLG-9349. All the thickener ingredients are commercially available and the manufacturing procedure was greatly simplified by this choice of ingredients. The corresponding grease made with DC XF 258 Silicone Fluid was designated MLG-9360 and comes closest to meeting the proposed requirements for a grease for the temperature range from -65° to 450°F. Arylurea grease of Harcoflex 600 (polyester) is the best choice for a grease for use from -65° to 350°F with good lubricity; bearing performance at 350°F averaged about eight times longer than that obtained with MIL-G-3278 greases. Pteridine derivatives were the most promising compound uncovered during this period for thickeners for use in greases in excess of 450°F and as high as 700°F. An anti-oxidant, MLO-55-535, was at least as good as phenothiazine or dilauryl selenide in ester greases. An anti-wear agent, MLO-55-499, in silicone greases was effective in reducing wear in the Navy gear-wear test, but not enough to pass the requirements under the ten-pound-load conditions. The fluid continues to be the limiting component in the development of a grease with good lubricity for the temperature range from -65° to 450°F and above.

WADC TR 53-83 Pt V  
ASTIA Document No. AD 118219  
OTS Release

April 1957

**SUBJECT:** DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASE  
THICKENERS

**INVESTIGATOR:** John C. Goossens

**CONTRACT:** AF 33(038)-23687

**CONTRACTOR:** Standard Oil Company (Indiana)

**ABSTRACT:** In this work directed toward the development of new grease thickeners operable at temperatures above 450°F and as high as 700°F, a variety of materials have been tested. Those thickeners which produced greases in DC 550 Silicone Fluid in less than 30% concentration and withstood preliminary screening tests were further tested in the ABEC-NLGI Bearing Tester.

The general types of materials tested as high temperature thickeners were organo-metal compounds, polymers and polar organic compounds. Selection of materials for test was based on thermal stability, ease of preparation and ability to prepare the substance in small particle size. Of the many materials tested, those which have shown the greatest promise are substituted pteridines, urea derivatives and a polymer of melamine with dimethylsulfoxide.

As they presented themselves, a few additives for improving the lubricity and load carrying capacity of silicone fluids were tested in the Navy Gear Wear Test and the Shell Four Ball Wear Test. None of the tested additives were notably effective. Thus a number of promising high temperature grease thickeners are available while the fluid continues to be the major limiting factor in the development of a lubricating grease for use as high as 700°F.

WADC TR 53-293 Pt 6

April 1956

SUBJECT: HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS  
Part 6. Evaluation of Additives Mechanism and Radiochemical Studies

INVESTIGATOR: James W. Cole, Jr.  
Donald R. Campbell

CONTRACT: AF 33(038)-22947

CONTRACTOR: University of Virginia

ABSTRACT: The object of this work is the evaluation of promising additives and the determination of their mechanism of action as anti-oxidants in non-petroleum base hydraulic and lubricating fluids at temperatures in the range 400° to 700°F. This report describes the effects of structure and concentration of the additives and the effects of test specimens of copper, silver, aluminum, titanium, magnesium, and steel in diester, silicate, disiloxane-silicone, silicone, and phosphate base fluids. Mechanisms of oxidation and anti-oxidant activity have been postulated with radioactive sulphur being used as a tracer in some of these quantitative studies.

Phenothiazine and its derivatives showed some effectiveness as anti-oxidants in a representative di-ester sebacate fluid over the temperature range 400° to 500°F. Several other additives, including aryl amines and selenium derivatives, appear promising in the higher temperature range. Magnesium, copper and iron were most severely attacked with titanium, aluminum and silver generally being unaffected.

No additive system appears to meet specifications for use in mixed C<sub>6</sub>-C<sub>8</sub> silicates, blended disiloxane-silicone, and DC silicone 550 although some had anti-oxidant activity.

Additives showing promise for use in a G. E. silicone in the range 500° to 700°F are p,p'-dioctyldiphenylamine, phenyl-~~alpha~~-naphthylamine and N,N'-di-2-naphthyl-p-phenylenediamine.

Mechanism studies were performed in a special apparatus using small amounts of fluid under controlled conditions of flow, temperature and pressure. Radiochemical studies of S<sup>35</sup> phenothiazine in di-(2-ethylhexyl) sebacate show the rate of formation of insoluble matter and indicate the relative contribution of the fluid and additive in forming sludges and in attacking metal surfaces.

WADC TR 53-293 Pt 7  
ASTIA Document No. AD 110728  
OTS Release

January 1957

SUBJECT: HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS  
Part VII Evaluation of Antioxidants in Synthetic Fluids  
INVESTIGATOR: James W. Cole, Jr.  
CONTRACT: AF 33(616)-3234  
CONTRACTOR: University of Virginia  
ABSTRACT: The object of this work is the evaluation of selected additives as antioxidants and oxidation inhibitors for non-petroleum base lubricating and hydraulic fluids in the range 400° to 700°F. The emphasis in this report is on the further examination of selected amines in methyl phenyl silicones, chlorophenyl silicones, and a tetrakis-p-dodecyl silane in the presence of aluminum, silver, copper, stainless steel and titanium. Acridines, dipyridyl amines and aryl phenylene-diamines showed considerable promise as inhibitors of increases in viscosity arising from oxidative attacks. Variations in the relative effectiveness of other amines examined do not allow the establishment of definite relationships between structure and antioxidant activity at the upper temperatures.

Some studies with selected additives in a pentaerythritol ester and in bis-(1-methyl cyclohexylmethyl) sebacate in the range 400°-500°F showed the same general oxidative phenomena as previously described for bis-(di-2-ethylhexyl) sebacate. Experiences are described with five chlorophenyl phosphate fluids in the range 600°-700°F. This type has considerable inherent stability to oxidation in the absence of additives and metals. No additive was found which inhibited oxidation in the presence of metals.

WADC TR 53-337 Pt 3

May 1956

SUBJECT: POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS  
INVESTIGATOR: Charles F. Raley, Jr.  
CONTRACT: AF 33(616)-276  
CONTRACTOR: Southwest Research Institute

WADC TR 53-373 Sup 4

**ABSTRACT:** The program of investigation of the class of aryl phosphate esters was continued. These materials possessed the most promising high-temperature properties of those examined. The aryl groups found to be the most thermally stable were phenyl, fluoro- and chlorophenyl, m-trifluoro-methylphenyl, 4-biphenyl, dimethylphenyl, 5-indanyl and naphthyl. The simple phosphate structure was found to be quite stable. Pyrophosphates had the drawbacks of mediocre thermal and hydrolytic stability, although apparently possessing good oxidative stability.

Compositions prepared by reacting two or three different phenols with the proper amount of  $\text{POCl}_3$  were also investigated. These compositions exhibited markedly lessened tendency to crystallization. Many of the compositions possessed properties almost identical to pure compounds having equivalent phenol proportions, and had the advantage of greater ease of preparation.

A total of nine fluids were prepared which possessed a liquid range of at least  $-20$  to  $800^\circ\text{F}$ . Many of the compounds and compositions prepared, including others not meeting this liquid range, are considered to have potential use value.

Several generalizations were drawn from the results of the over-all program. It was found that thermal and oxidative stability do not necessarily go together. Strongly polar substituents on the aromatic ring adversely affect hydrolytic stability. Two or more polar groups on a monocyclic ring adversely affect the thermal stability; one polar group on a bicyclic ring adversely affect the thermal stability. A marked effect on such physical properties as boiling point, melting point, viscosity, etc., is observed, depending on the nature of the substituent groups. The more compact molecules generally have the greatest tendency to crystallize, with boiling point increasing with the molecular weight.

WADC TR 53-426 Pt 3  
ASTIA Document No. AD 97236

August 1956

**SUBJECT:** ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS

**INVESTIGATORS:** Henry Gilman  
Richard D. Gorsich

**CONTRACT:** AF 33(616)-94

**CONTRACTOR:** Iowa State College

**ABSTRACT:** The purpose of this investigation is to synthesize and evaluate organometallic and organometalloidal compounds as base stock materials for high-temperature lubricants and hydraulic fluids. Preliminary screening

has been extended and has shown that some of the more promising types are cyclic in nature where silicon or germanium is part of the heterocycle. In addition, the biphenyl group, the phenoxyaryl groups, the long-chained alkyl groups and the haloaryl groups are very promising.

New syntheses have been developed for a number of these types, and these are described in the experimental section. There is also included, in the experimental part, a table which outlines the preliminary screening of forty-one compounds.

WADC TR 53-466 Pt II  
ASTIA Document No. AD 97318

September 1956

SUBJECT EVALUATION OF DRY FILM LUBRICANTS

INVESTIGATOR: Bernard Rubin

ABSTRACT: A large number of commercial films were evaluated by the previously reported Falex Test procedure and wide variation in wear endurance life noted. The nature of failure pattern and effect of load are noted for some of these films. Certain films showed very good life in the repeatable 30 minute test procedure. Titanium disulfide in a resin binder had poor fraction and endurance properties. Tung-Lube, a proprietary friction reducer, showed some promise and should be evaluated further.

Preliminary data at elevated temperatures indicate that high temperature static soak (to 500°F) and subsequent test at room ambient temperature is much less severe on endurance life than evaluation at 225°F. Endurance life of films on line contact specimens (block and cylinder) is shown to vary inversely with speed and load. However, at light loads sliding velocities of 300 to 400 feet per minute for significantly useful periods of time under unidirectional motion have been observed for a representative dry friction reducing film

WADC TR 54-339  
ASTIA Document No. AD 110426  
OTS Release

October 1956

SUBJECT: DETERMINATION OF THE MECHANISM OF THE INCREASE OF VISCOSITY  
OF ORGANOSILICON COMPOUNDS AT HIGH TEMPERATURES

INVESTIGATOR: Edward E. Ryskiewicz

CONTRACT: AF 33(616)-168

CONTRACTOR: Stanford Research Institute

ABSTRACT: A study of the oxidation of the tetrapentoxysilanes at 120°C (248°F) has revealed an apparent correlation between oxidation rate and chemical structure.

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Antioxidants could not be studied effectively at 120°C (248°F) because of the prolonged protection afforded by all the compounds studied. Ionol in a 0.1% concentration protected tetra (2-pentoxy)silane against oxidation at 120°C (248°F) for 408 hours.

Hydrolysis of the tetrapentoxysilanes was shown to be markedly affected by products which could form during oxidation. An apparent relation between chemical structure and rate of hydrolysis has been found for the tetrapentoxysilane isomers.

WADC TR 54-464 Pt 2

June 1956

SUBJECT: DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES  
INVESTIGATOR: Josef J. E. Schmidt  
John R. Hobough  
Francis S. Bonomo  
CONTRACT: AF 33(616)-2204  
CONTRACTOR: Denver Research Institute  
ABSTRACT: General properties and methods for the identification and determination of components of synthetic lubricants and base-oils and their additives are presented and discussed.

Included in these compounds are dibasic acid esters, silicate esters, and silicone oils, and such additives as antioxidants. Analytical procedures employing column and paper partition chromatographic techniques are presented for the identification and separation of different groups of components, such as dibasic acid esters from silicate esters and silicone oils as well as different dibasic esters from each other, along with methods for the paper chromatographic identification and separation of antioxidants in synthetic lubricants.

WADC TR 54-464 Pt 3  
ASTIA Document No. AD 118215  
OTS Release

April 1957

SUBJECT: DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES  
INVESTIGATOR: Francis S. Bonomo  
CONTRACT: AF 33(616)-3336  
CONTRACTOR: Denver Research Institute  
ABSTRACT: Methods for the separation, identification, and determination of certain components of synthetic greases and synthetic lubricants are presented and discussed. Included in these components are such base-oils as dibasic acid esters, silicate esters, disiloxanes, and silicone oils, and selenide and phenol type antioxidants.

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Analytical chromatographic procedures employing column partition, paper partition, and adsorption techniques for the separation and identification of different groups of components are presented and discussed. Column partition chromatography is shown to be a useful means for isolating high molecular weight dibasic acid esters from silicate esters, disiloxanes, and silicone oils. Adsorption chromatography is employed for the separation and concentration of antioxidants from base-oils, for the removal of impurities from lubricants, and for qualitative comparisons of lubricants of known compositions.

Qualitative and quantitative procedures for the detection and determination of selenium-containing antioxidants are presented. Attempts to detect phenol-type antioxidants by volatilization were unsuccessful.

Preparation of the laboratory manual, an outline of the analytical methods in the manual, and a list of ASTM methods included in whole or in part in the manual are discussed briefly. The manual itself, resulting from two and one-half years' research for WADC under Contracts AF 33(616)-2204 and AF 33(616)-3336, is Part IV of this report.

WADC TR 55-30 Pt 4  
ASTIA Document No. AD 110688

October 1956

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS  
INVESTIGATOR: E. Erwin Klaus  
Merrell R. Fenske  
CONTRACT: AF 33(616)-2851  
CONTRACTOR: Petroleum Refining Laboratory, Pennsylvania State University  
ABSTRACT: This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids, jet engine lubricants, and jet fuels for the Air Force. Primary emphasis for the period covered by this report has been placed on the study of the high temperature aspects of these fluids developments.

Hydraulic fluid developments have been focused on fluids suitable for use at 500° to 700°F. for limited times in sealed hydraulic systems. Tests have been developed for a basic study of viscosity, thermal stability, oxidation stability, and lubricity in this desired temperature range. The high temperature properties of 10 chemical classes of fluids have been surveyed. Additional efforts have been concentrated on the study of chlorine-containing silicones, hydrocarbons, esters, and chlorinated aromatic hydrocarbons for application to these high temperature hydraulic systems.

Jet engines lubricants are needed for operation at 500° and 600°F bulk oil temperatures and up to 700°F. hot spot temperatures. In addition to the data obtained in the hydraulic fluid study, emphasis has been

placed on more severe lubricity, oxidation, and corrosion requirements. Again emphasis has been placed on the same four types of compositions. Lubricity, oxidation, and corrosion studies over the range of 500° to 700°F. are presented. Attention has been focused on the best utilization in a lubricant system of the properties obtainable in this temperature range.

Techniques for the measurement of jet fuel dirtiness have been studied. Methods of improving jet fuel dirtiness by additives and other treatments have been investigated.

Some additional miscellaneous duties in the form of fluid formulations, distribution of standard fluid samples and Laboratory reports have been performed at the request of the Wright Air Development Center.

WADC TR 55-102 Pt III  
ASTIA Document No. AD 110701

January 1957

SUBJECT: GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS

INVESTIGATOR: John B. Accinelli

CONTRACT: AF 33(616)-2443

CONTRACTOR: Shell Development Company

ABSTRACT: The report summarizes the final work done on grease lubricated anti-friction bearings at speeds of  $1.0 \times 10^6$  DN and higher. The work was performed at the Shell Development Company on two 20 mm ultra high speed ball bearing rigs (Rig No. AF-2443-1 and -2) designed and built by this company for the Air Force. Work with grease at  $1.0$  to  $1.2 \times 10^6$  DN suggests that the interplay of mechanical factors such as rotor dynamic unbalance, bearing type, size and precision, load alignment, bearing-to-housing fits, etc., will affect performance more than changes in grease properties. Grease lubrication at these ultra high speeds appears to be affected considerably more by mechanical factors than is oil-air mist lubrication, indicating that grease lubrication must be inherently more marginal. One test at  $1.0 \times 10^6$  DN with a grease meeting MIL-G-3278 ran well for 109 hours, thus demonstrating that under the proper conditions satisfactory long term lubrication with grease is possible.

With all greases, operation of the rig at 30,000 rpm was significantly better than operation at 50,000 rpm. A grease containing silicone oil was poor at both speeds.

WADC TR 55-190 Pt. III  
ASTIA Document No. AD 118236  
OTS Release

April 1957

SUBJECT: DEVELOPMENT AND EVALUATION OF A GREASE FOR -100°F. TO +350°F.  
INVESTIGATOR: Ernest W. Nelson  
Warren W. Woods  
William P. Scott  
CONTRACT: AF 33(616)-2364  
CONTRACTOR: Continental Oil Company

ABSTRACT: In the work directed toward the development of an aircraft grease suitable for use over a wide temperature range, emphasis has been placed on the utilization of certain hexa alkoxy disiloxane fluids as base oils. The thickener was limited to colloidal calcium carbonate coated through chemical interaction with the calcium salt of 3-methyl glutaric acid. Formulations using the base oil hexa (2-ethyl butoxy) disiloxane show promise of utility over a -100°F. to +325°F. temperature range. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane show promise of utility over -100°F. to +350°F. This fluid was not available in large quantities until the last quarter of the contract period. Time has permitted the preparation and evaluation of only a limited number of hexa (2-ethyl hexoxy) disiloxane greases. These greases perform in the range of 450 to 600 hours at 350°F. in the high speed testers, providing pure oxidation inhibitor (p,p'-dioctyl-diphenylamine) is used. The pure grade is not commercially available at this time. The use of commercial grade inhibitor limits performance to around 150 hours at 350°F.

WADC TR 55-240 Pt II  
ASTIA Document No. AD 97282

September 1956

SUBJECT: BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES  
INVESTIGATOR: John J. Chessick  
Albert C. Zettlemoyer  
CONTRACT: AF 33(616)-2440  
CONTRACTOR: Lehigh University  
ABSTRACT: Physical properties of several oils and surface characteristics of a variety of thickening agents were measured in order to index the nature and extent of the vehicle-thickener interface in non-soap grease dispersions. These properties of the oils and solids were correlated with the behavior of grease systems formulated from them. Modification of the interfacial region by water and polar organic additives was also investigated.

The combined results of these studies have led to some interesting conclusions regarding the mechanism of flocculation with consequent gel formation of polar and nonpolar solids dispersed in grease vehicles. The relatively low area polar solids used in this work require the presence of small amounts of water in the grease system for flocculation to occur in nonpolar vehicles. These polar solids, at least in the quantities used in this work, would not be effective thickeners in pure liquids or in liquids which dissolve or are miscible with large quantities of water. On the other hand, the nonpolar

thickeners when dispersed in a vehicle do not need water to flocculate into a gel structure. These nonpolar materials were shown to be effective thickeners in both polar and nonpolar liquids. A proposed mechanism to explain the flocculation of these solids is based on the reduction in free surface energy brought about by the flocculation.

In addition, the mechanism of additive action has been explained qualitatively in terms of the mechanism of flocculation of these solids in the simple grease systems.

A variety of experimental approaches were used to gain a better understanding of grease structure. These included electrokinetic and conductance measurements and low angle X-ray scattering. X-ray scattering techniques have proved to be the most rewarding; indeed, marked diffraction maxima have been found corresponding to spacings of the order of 100 to 2000 Å units for copper phthalocyanine thickened greases

WADC TR 55-385  
ASTIA Document No. AD 110664  
OTS Release

March 1957

SUBJECT: HIGH TEMPERATURE EVALUATION OF ANTIOXIDANTS IN DIESTER BASE FLUIDS  
INVESTIGATOR: Howard D. C. Hill  
ABSTRACT: This study was initiated to evaluate various organo-selenium, organo-sulfur, and organo-nitrogen additives as oxidation inhibitors in diester base fluids. Samples of these materials were submitted to oven storage at 350°F for a period of eight (8) days after which evaporation losses, change in viscosity, and sludge content was determined. A temperature of 350°F was selected as representing the upper temperature limit of a -100° to 350°F grease requirement for which the additives were evaluated.

The results of the tests discussed herein indicate that, of the three groups of compounds evaluated, the selenium-containing compounds provided the best oxidation inhibitors from the standpoint of low evaporation losses and low viscosity change.

WADC TR 55-449 Pt 1  
OTS Release

April 1956

SUBJECT: MICRO LUBRICANT TEST METHODS VISCOSITY-NEUTRALIZATION NUMBER  
INVESTIGATOR: John B. Christian Arthur L. Miller  
Vernon A. Lauer Harry M. Schiefer, 1/Lt  
ABSTRACT: This study was initiated to develop miniaturized tests for petroleum, petroleum products, and related materials. Procedures for determining neutralization number, and viscosity are described and their correlation with macro tests provided.

WADC TR 53-373 Sup 4

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The small scale test techniques discussed herein are considered as satisfactory as their full scale counterpart for their intended purpose.

WADC TR 55-449 Pt 3  
OTS Release

May 1956

SUBJECT: MICRO LUBRICANT TEST METHODS Part 3. Corrosion and Oxidation-Separation of Oil From Lubricating Greases---Corrosiveness of Greases and Oils

INVESTIGATOR: John B. Christian

ABSTRACT: This report contains the following test methods: Corrosion and Oxidation, Separation of Oil from Lubricating Greases, and Corrosiveness of Greases and Oils. These test methods are designed for the micro analysis of petroleum, petroleum products, and related materials. They were arrived at through the modification of existing test procedures which require greater quantities of test samples.

WADC TR 55-449 Pt 4  
ASTIA Document No. AD 110587  
OTS Release

November 1956

SUBJECT: MICRO LUBRICANT TEST METHODS Part 4 Evaporation Loss of Lubricating Greases and Oils, Viscosity of Lubricants at -65°F, and Foaming Characteristics of Crankcase and Aircraft Engine Lubricating Oils

INVESTIGATOR: John B. Christian

ABSTRACT: This report describes test methods for the determination of evaporation loss of lubricating greases and oils, viscosity of lubricants at -65°F., and the foaming characteristics of crankcase and aircraft engine lubricating oils. All procedures were arrived at through the comparison of the micro test results with results obtained through the use of existing test procedures.

WADC TR 55-449 Pt 5  
ASTIA Document No. AD 118048  
OTS Release

February 1957

SUBJECT: MICRO LUBRICANT TEST METHODS Part 5. High Temperature Viscosity

INVESTIGATOR: John B. Christian

ABSTRACT: This report introduces a small scale method of analysis for the determination of viscosity at high temperatures (550°-700°F). Its application is especially important in instances where only minute quantities of

sample are available. The small scale replica of the modified Ostwald Viscometer employing 1.5 millimeters of sample was used in the test method described.

A bath solution composed of the nitrates of lithium, potassium, and sodium has been substituted for the usual oil bath and has proved to be satisfactory.

WADC TR 55-475  
OTS Release

May 1956

SUBJECT: FOAMING CHARACTERISTICS OF AIRCRAFT OILS  
INVESTIGATOR: George Baum  
ABSTRACT: The foaming characteristics of several lubricating oils were studied in connection with power plant and propeller installation difficulties ascribed to oil foaming. Wide variations in quantity and collapse time of foam formed during aeration were observed in laboratory tests. Chemical additives of silicone and sorbitan type proved effective as heavy petroleum oil defoamers. No really effective defoamers for light oils were noted. A laboratory test which gives a sharper delineation between a group of light oils than the present ASTM test has been studied and may with sufficient refinement provide a better index of foaming tendency of oils than the present test.

WADC TR 56-1  
ASTIA Document No. AD 110702  
OTS Release

January 1957

SUBJECT: ALASKAN TEST SITE OIL EXPOSURE PROGRAM  
INVESTIGATOR: Robert J. Benzing  
ABSTRACT: A program was undertaken to determine the effect of prolonged outdoor unheated shed storage on the pour points of several specification oils of mineral oil and diester base stock origin. Permanent increases in pour point following prolonged cyclic low temperature storage had previously been reported for automotive and industrial oils. Qualitative daily visual observation and monthly pour point data on ten mineral oils and four diester oils covering two years of observation in Fairbanks, Alaska, are reported.

Petroleum oils, in general, showed increases in pour points after storage while the synthetic base oils had stable pour points. The conclusion drawn was that with the increase of use of synthetic oils, no problem should be experienced in low temperature use after storage.

December 1956

SUBJECT: THE PREPARATION AND PROPERTIES OF SOME FLUORINE-CONTAINING  
DIESTERS

INVESTIGATOR: Joseph F. O'Brien, Capt. Jack V. Fenner, 1/Lt  
Robert Filler George Rappaport

ABSTRACT: The preparation and properties of six different types of fluorine-containing diesters for possible use as high temperature lubricants and hydraulic fluids are described. Both dicarboxylic acid diesters and glycol esters were prepared from fluorine-containing mono- and dicarboxylic acids, alcohols and glycols. Diesters derived from fluorine-containing acids and hydrocarbon alcohols or glycols were readily prepared by direct esterification. Diesters derived from fluorine-containing alcohols or glycols and hydrocarbon acids or fluorine-containing acids were prepared from the acid chloride and alcohol or glycol. The fluorine-containing diesters most resistant to hydrolysis were derived from the fluorine-containing alcohols or glycols and the hydrocarbon acids. The boiling point, refractive index, and surface tension of the fluorine-containing diesters were lower than those of their hydrocarbon analogs, whereas the density and absolute viscosity were higher. The infra-red spectra of the diesters were recorded and the effect of fluorine atoms on the ester carbonyl frequency is described.

WADC TR 56-175  
OTS Release

May 1956

SUBJECT: STARTING AND RUNNING LOW TEMPERATURE TORQUES

INVESTIGATOR: Bernard Rubin

ABSTRACT: Low temperature (-65°F) starting and running torque data for greases are reported in size 104, 204, and 306 bearings using a procedure under investigation by the ASTM Technical Committee G on Lubricating Grease. The investigation with the 204 bearing was a part of the cooperative effort with other laboratories within the ASTM group working towards the standardization of a low temperature torque procedure. Volume of grease in the bearing could not be directly correlated with starting and running torque.

Recommendations are made for improvement of test procedure for specification purposes.

WADC TR 56-177  
ASTIA Document No. AD 97138  
OTS Release

July 1956

SUBJECT: POLYARYLUREA GREASES

WADC TR 53-373 Sup 4

INVESTIGATOR: D. T. Kjerland, 2/Lt  
ABSTRACT: Arylurea compounds prepared from aromatic isocyanates or aromatic diisocyanates and an aromatic amine or combination of aromatic amines have in the last three years been shown to be good grease thickening agents having high temperature (500°F) stability. In this report, preliminary data are presented on polyarylurea thickening agents prepared from aromatic diisocyanates and aromatic diamines.

Initial data show that polyarylureas have much better thickening ability than the simple arylureas with equivalent heat stability.

WADC TR 56-224  
ASTIA Document No. AD 97291

September 1956

SUBJECT: HIGH TEMPERATURE LUBRICANT STUDIES  
INVESTIGATOR: E. Erwin Klaus  
Merrell R. Fenske  
CONTRACT: AF 33(616)-2851  
CONTRACTOR: Petroleum Refining Laboratory, Pennsylvania State University  
ABSTRACT: Basic information on the behavior of hydraulic fluids and lubricants is needed at temperatures above 400°F. if successful operation of engines and hydraulic systems is to be achieved at high temperatures. It is necessary first to develop reliable test procedures and techniques covering the measurement of viscosity, lubricity, corrosion, and oxidation and thermal stability at temperatures of 500° to 700°F. Several fluids were evaluated in this manner. These fluids are representative of the different chemical classes, such as silicones, silicates, hydrocarbons and mineral oils, diesters, and halogenated aromatic hydrocarbons.

It was found that the property differences between the various types of fluids at 500° to 700°F. were much narrower than exist in the 200° to 350°F range. Data are presented showing the inadequacy of extrapolating 200° to 350°F. data as a means of predicting performance at 500° to 700°F. The behavior of fluids and lubricants in the 200° to 350°F. range has been quite well defined in recent years for the materials used in this study. However, there is a paucity of information at 500° to 700°F. This paper attempts to remedy somewhat this situation.

WADC TR 56-229  
ASTIA Document No. AD 110597  
OTS Release

November 1956

SUBJECT: STUDY OF RUST-INHIBITING COMPOUNDS IN SYNTHETIC GREASES BY  
THE HUMIDITY CABINET METHOD



INVESTIGATOR: H. Schwenker  
H. D. C. Hill  
R. J. Benzling

SUBJECT: A series of rust-inhibitors were investigated for their effectiveness in a Lithium 12 hydroxystearate-di-2(ethyl-hexyl) sebacate grease and in a Bentone 34-dipropylene-glycol-dipelargonate grease by means of the Humidity Cabinet Test. Mechanical stability tests were made on the soap greases and electronmicrographs taken of several of the test greases. Sulfonates and amines were the most effective inhibitors in the soap-diester greases. The amount of inhibitor used was also found to be critical with regard to the effectiveness of certain additives. Mechanical stability of the soap-diester type greases was not affected by the various rust-inhibitors. Some effect on the unworked penetrations of the soap-diester greases when different inhibitors were used was noted. Electronmicrographs of the soap-diester greases revealed a difference in the dispersion and growth of the soap fibers when in the presence of certain inhibitors. However, no significant effect on the consistency of these greases was noted.

The Bentone 34-dipropylene-glycol-dipelargonate greases were not particularly susceptible to rust-inhibitors of any type. The best results obtained were with sulfonates and dibutyl ammonium oleate. However, these inhibitors provided only borderline protection at most. No effect on the mechanical stability of these greases by the use of rust-inhibitors was noted.

WADC TR 56-254  
ASTIA Document No. AD 97184

August 1956

SUBJECT: MINERAL OILS AS HIGH TEMPERATURE FLUIDS AND LUBRICANTS  
INVESTIGATOR: E. Erwin Klaus  
Merrell R. Fenske  
CONTRACT: AF 33(616)-2851  
CONTRACTOR: Petroleum Refining Laboratory, Pennsylvania State University  
ABSTRACT: In a survey of fluids and lubricants for use at 400° to 700°F., certain types of mineral oils were found to be among the most promising materials studied. While synthetic lubricants have better performance characteristics than most mineral oils and hydrocarbons in the moderate temperature range (200° to 400°F.), they do not possess this margin of superiority in the high temperature range (400° to 700°F.). Certain synthetics have better viscosity-volatility and viscosity-temperature characteristics than hydrocarbons.

Some mineral oils and diesters of the Spec. MIL-L-7808 type have good lubricity properties at high temperatures. Using a hydraulic pump and a laboratory wear tester, it has been found that there are lubricity additives that are still effective at 500° to 700°F. in mineral oils and diesters.

Mineral oils and certain types of hydrocarbons have better thermal stability and oxygen tolerance than many synthetics at 600° to 700°F. The concept of oxidation stability, or inertness toward oxygen, is not normally applicable at these temperatures. Oxidation rate and oxygen tolerance are better criteria for judging oxidative deterioration at 500° to 700°F.

It appears that some additives and additional refining techniques (such as hydrogenation) will be beneficial in improving the oxidation behavior of mineral oils and certain other hydrocarbons at moderate and high temperatures.

The thermal and oxidative components of high temperature coking on metal surfaces are demonstrated in a controlled atmosphere panel coker. It may be necessary to control the atmosphere in engine parts requiring lubrication at 500° to 700°F. because of the possibility of spontaneous ignition of the lubricant or of fragments from its decomposition.

In a lubrication system where the oil is used for a limited time and then discarded (perhaps to the burners) it is necessary to know the effect of contaminating the fresh oil with oxidized or deteriorated oil. Data have been obtained indicating that this effect need not be serious with esters and mineral oils at 500°F.

WADC TR 56-262

October 1956

ASTIA Document No. AD 110422

SUBJECT: OPERATING INSTRUCTIONS FOR GEAR AND SPLINE LUBRICANT TESTER  
INVESTIGATOR: Charles W. Sauter  
CONTRACT: AF 33(616)-2967  
CONTRACTOR: Western Gear Corporation  
ABSTRACT: A universal gear lubricant tester has been constructed to evaluate developmental high temperature gear lubricants under simulated flight conditions. This report gives the operating instructions for such a tester operating at speeds up to 30,000 revolutions per minute and temperatures up to 800°F

WADC TR 56-275

March 1957

ASTIA Document No. AD 118110

OTS Release

SUBJECT: RECLAMATION OF SYNTHETIC BASE OILS  
INVESTIGATOR: Richard W. Sneed  
H. M. Schiefer, 1/Lt

**ABSTRACT:** The feasibility of the reclamation of synthetic base oils, specifically of the diester type, has been investigated. Various filtration, percolation, and distillation techniques have been employed in an attempt to clean used oils to such an extent as to make them reusable, either as an aircraft gas turbine engine lubricant or as a plasticizer grade ester. It has been determined that hydraulic fluid used for flushing alternator drives can be filtered and used again instead of being discarded as is the present practice in depots. Preliminary studies of reclamation procedures for used engine oils indicate that full scale reclamation is possible. However, considerable work would have to be done to ascertain the efficiency and economy of the reclamation operation and also the problems involved with full scale operation of engine with reclaimed oil.

WADC TR 56-370  
ASTIA Document No. AD 110411

October 1956

**SUBJECT:** RESEARCH IN HIGH TEMPERATURE BEARING LUBRICATION IN THE ABSENCE OF LIQUID LUBRICANTS  
**INVESTIGATOR:** Charles H. Bailey  
Stanley S. Sorem  
**CONTRACT:** AF 33(616)-2999  
**CONTRACTOR:** Shell Development Company  
**ABSTRACT:** The applicability of the "protective atmosphere" system to the operation of high temperature alloy rolling element bearings at temperatures from 600°F to 800°F is being investigated. This system involves surrounding the operating bearing with a reducing atmosphere consisting of a mixture of organic vapor and a volatile extreme pressure additive in air. The reducing environment prevents the formation of destructive abrasive metal oxides. Reactions of the extreme pressure additive and the organic vapor with or on the metal surfaces results in the formation of films which provide the necessary "lubrication" for high temperature operation.

Results of this investigation to date show that the "protective atmosphere" system is applicable to bearings constructed of ferrous alloys. Bearings with tool steel balls and races and steel cages appear best suited. The desired films cannot be formed on metals of low oxidation potential such as bronze or silver nor on the oxidation resistant alloys such as the stainless steels. The contribution of the organic portion of the atmosphere to the protective film formation has only recently been recognized. Exploitation of this discovery may lead to further improvements in "protective atmosphere" composition.

Best results thus far have been obtained with a "protective atmosphere" consisting of a mixture of air and JP-4 fuel vapor in a weight ratio of 8 to 1. The JP-4 used contained about 0.2% sulfur. In this atmosphere a size 206 all ferrous metal bearing has been operated 100 hours at

10,000 rpm and 700°F without appreciable wear or other indications of bearing deterioration. The limits of the "protective atmosphere" system with respect to bearing temperature, speed and load are yet to be determined.

WADC TR 56-370 Pt II  
ASTIA Document No. AD 118121

March 1957

SUBJECT: RESEARCH IN HIGH TEMPERATURE BEARING LUBRICATION IN THE  
ABSENCE OF LIQUID LUBRICANTS  
INVESTIGATOR: Charles H. Bailey  
Stanley S. Soren  
CONTRACT: AF 33(616)-2999  
CONTRACTOR: Shell Development Company  
ABSTRACT: The "protective atmosphere" system for operation of high  
temperature alloy rolling element bearings at temperatures from 600°F to 800°F  
is being investigated. This system involves surrounding the operating bearing  
with a special reducing atmosphere. This consists of a "rich" mixture of  
organic vapor and air with small additions of volatile film forming compounds.  
The reducing environment prevents the formation of destructive abrasive metal  
oxides. Reactions of the additive and organic vapor with or on the metal  
surfaces results in the formation of films which provide the necessary "lub-  
rication" for high temperature operation.

Phase I work on mechanisms and atmosphere compositions was continued using size 206 bearings operating under light load at 10,000 RPM. The upper temperature limit of a specific atmosphere composition was found to be 800°F. Possible reasons for the limit are given and means of increasing it are discussed. A group of oxygen and nitrogen containing compounds naturally occurring in JP-4 (approximately 1%w) were found to beneficially affect the nature of the lubricating films formed, thus leading to superior "protective atmosphere" operation. Several representative compounds from this group have been tested individually and of these m-toluidene was outstandingly good.

Phase II work, outlining the load, speed, and life characteristics of bearings operated in a "protective atmosphere" was started using size 209 bearings operating at various speeds and loads. A bearing operated at 700°F under 2/3's of catalog load rating successfully completed 33 hours. Wear was appreciable during this test but compromises in both the mechanical installation and atmosphere composition were necessary in order to work with available bearings. These may have affected the wear results.

WADC TR 56-375  
ASTIA Document No. AD 110585  
OTS Release

January 1957

SUBJECT: WEAR STUDIES WITH TITANIUM

INVESTIGATOR: Robert J. Benzing  
Arthur N. Damask

ABSTRACT: A study of the wear of titanium using typical oils was made on the Falex Wear Tester and the Shell Four-Ball Wear Tester. The purpose of this study was to provide data for comparison with other studies dealing in friction coefficients and wear of specimens in non-standard lubricant testers. A mineral oil, diester, silicate ester, silicone, and halogenated hydrocarbon were the oils studied. C-130-AM, Ti-150A, and 3Al-5Cr alloys were used and both cyanided and uncyanided surfaces are included in the program.

WADC TR 56-430 Pt I  
ASTIA Document No. AD 110644  
OTS Release

December 1956

SUBJECT: THE EFFECTS OF NUCLEAR RADIATION ON MILITARY SPECIFICATION GREASES

INVESTIGATOR: William L. R. Rice, 1/Lt

ABSTRACT: A portion of the Air Force effort toward the development of nuclear radiation resistant lubricants is devoted to an evaluation of the effects of gamma radiation on available specification and non-specification greases. Data are presented on the effects of gamma radiation on forty-seven greases. Many of the greases tested appear to be satisfactory for use after exposure to about  $1 \times 10^8$  roentgens, the screening dosage used for these studies.

WADC TR 56-475  
ASTIA Document No. AD 110634  
OTS Release

December 1956

SUBJECT: EVALUATION TESTS OF ARYLUREA-SILICONE GREASES IN AIRCRAFT EQUIPMENT

INVESTIGATOR: Herbert Schwenker

ABSTRACT: Several types of arylurea-silicone greases have been evaluated in various types of test equipment. Some of the tests were conducted with actual aircraft components, while other tests consisted of mock-ups of various aircraft systems.

Arylurea-silicone greases have good performance in the following high temperature applications: airframe oscillatory bearings, electric motor bearings, low speed roller bearings, instrument bearings.

Arylurea-silicone greases did not function satisfactorily in screw jack actuator applications and in high speed ball bearings.

The type of application, equipment design, materials used in construction, and the surrounding environment all were found to be significant factors influencing the success or failure of arylurea-silicone greases to function as lubricants.

WADC TR 57-36 Pt I  
ASTIA Document No. AD 118277  
OTS Release

May 1957

SUBJECT: AIRFRAME LUBRICANTS  
INVESTIGATOR: Airframe Lubricants Group  
CONTRACT: AF 33(616)-2888  
CONTRACTOR: Coordinating Research Council, Inc.  
ABSTRACT: The annual report on Airframe Lubricants is a status report that reviews the activities of the various projects undertake to assist the Air Force in conducting research on the problems of mutual adaptation of airframe lubricants and equipment, with particular emphasis on the work done from June 1954 to December 1956. This report, covering seven projects in all (High Temperature Testing of Greases in Antifriction Bearings, Fretting Corrosion, Rust-Preventive Properties of Greases, Plain Bearings, Screw Thread and Gear Mechanisms, Bonded Solid-Film Lubricating Coatings, and Methods for Cleaning Bearings Prior to Use in Laboratory Tests), describes the scope and objective of each project, the work of the Laboratory and Service Evaluation Panels for each, the conclusions reached, and the plans under consideration for future work along the same lines.

WADC TR 57-113  
ASTIA Document No. AD 118246  
OTS Release

April 1957

SUBJECT: DETERMINATION OF THE SHEAR STABILITY OF VARIOUS AIRCRAFT LUBRICATING GREASES BY THE HIGH TEMPERATURE SHELL ROLL TESTER  
INVESTIGATOR: Donald T. Kjerland, 2/Lt.  
ABSTRACT: The shear stability of various high temperature lubricating greases was determined at temperatures up to 600°F by use of a Shell Roll Tester which was adapted for high temperature testing. The greases evaluated were of both soap and non-soap types and contained the following types of base fluids: silicone, pentaerythritol ester, diester, substituted m-terphenyl and biphenyl, and petroleum oil.

Bargraphs plotting time in the Shell Roll Tester at a given temperature and rotational speed versus the resultant grease consistency were prepared for each of the lubricating greases evaluated. Although the main

emphasis in this work was on the determination of the shear stability of a given grease at various temperatures, some testing was done in an effort to determine what effect, if any, rotational speed had on the change in grease consistency in this test.

The High Temperature Shell Roll Tester has been shown to be an effective method to determine the shear stability of a lubricating grease at high temperatures.

WADC TR 57-299  
ASTIA Document No. AD 118329  
OTS Release

May 1957

SUBJECT: NUCLEAR RADIATION RESISTANT LUBRICANTS  
INVESTIGATOR: William L. R. Rice, 1/Lt  
ABSTRACT: This report presents a general summary of the state of the art of nuclear radiation resistant lubricant development. Data are presented on the effects of gamma radiation on currently available fluids and lubricants, showing the limitations of each. Development of advanced materials is discussed, with a general outline presented of the approach taken by the major support contractors.

It is estimated that on the basis of information to date, conventional ester type lubricants should prove operable to at least  $1 \times 10^7$  roentgens of gamma radiation at temperatures from  $-65^\circ$  to  $350^\circ\text{F}$ . Improved ester types, of the aromatic ester or complex ester variety, with proper additives, are expected to give suitable performance up to about  $1 \times 10^8$  roentgens, operating in the temperature range  $-40^\circ$  to  $400^\circ\text{F}$  or higher. Selected mineral oils, usable from about  $0^\circ$  to  $400^\circ\text{F}$ , may possibly be reliable to about  $2$  to  $3 \times 10^8$  roentgens. The aromatic ethers, which are expected to be stable at temperatures well above  $400^\circ\text{F}$  (laboratory tests show thermal decomposition temperatures of  $700^\circ$  to  $800^\circ\text{F}$ ), have not been evaluated for radiation resistance, but based on similar types of aromatic compounds they should be stable in the range  $10^9$  to  $10^{10}$  roentgens. Much further work is required to verify this estimate.

A summary is presented of the future plans and major problem areas remaining in the fluid and lubricant development programs.

#### PLASTICS, STRUCTURAL

WADC TR 52-183 Sup 4  
ASTIA Document No. AD 97329  
OTS Release

September 1956

SUBJECT: ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN  
"PLASTICS FOR AIRCRAFT"  
INVESTIGATOR: Donald G. Coleman  
CONTRACT: AF 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1956 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Six technical reports issued during the fiscal are abstracted.

WADC TR 53-185 Pt 3  
ASTIA Document No. AD 97317

September 1956

SUBJECT: SUMMARY REPORT ON THE RAIN EROSION OF AIRCRAFT MATERIALS  
INVESTIGATOR: Roy R. Lapp  
Raymond H. Stutzman  
Norman E. Wahl  
CONTRACT: AF 33(616)-2758  
CONTRACTOR: Cornell Aeronautical Laboratory  
ABSTRACT: This report summarizes the data obtained on the relative rain erosion resistance, on the whirling arm tester, of a number of materials at subsonic speeds.

Several solvent type air dry coatings based upon neoprene were evaluated for conformance to Military Specification MIL-C-7439B. A modified Gaco N-79 neoprene coating was the only coating of the series tested that had satisfactory rain erosion resistance.

Polyurethane materials tested in the form of sheet or coatings applied to standard glass reinforced test specimens, while not as erosion resistant as neoprene, had very good erosion resistance and will be investigated more intensively.

Tests on fluorinated ethylene materials show that they possess a relatively low degree of rain erosion resistance.

Erosion tests were conducted on a number of white coating materials based upon alkyd enamels, vinyl, silicone, butyl, polysulfide and chlorosulfonated ethylene elastomers. Of all these white materials tested, coatings based upon Hypalon alone and combinations of white pigmented neoprene and polyacrylic rubber appeared to merit further study.



Of the outstanding glass and ceramic materials tested, Corning Glass 1710 took 45 minutes to erode while a ceramic body, Alsimag 576 of Alumina, lasted for approximately 420 minutes.

Comparison of current epoxy and polyester glass reinforced laminates indicates that the erosion resistance of standard test specimens of epoxy-glass laminates have four to five times the erosion resistance of similar laminates made with polyester resins.

Neoprene coated thin walled laminates, such as those used on hot air deiced radomes, were found to have very poor rain erosion resistance.

In collaboration with the National Bureau of Standards, studies on the mechanism of erosion and tests were conducted on a variety of different materials under specific conditions.

WADC TR 53-192 Pt 7  
ASTIA Document No. AD 118223

April 1957

SUBJECT: MECHANISM OF RAIN EROSION Part 7 Mechanism Studies on 1100 and 3003 Aluminum  
INVESTIGATOR: Olive G. Engle  
CONTRACT: AF 33(616)-53-9  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: A study of deformation marks made by impingement of steel spheres and of deforming lead pellets against 2S aluminum plates has proved helpful in analyzing the behavior of and the damage caused by waterdrops in collisions with 2S aluminum at both subsonic and supersonic relative impingement velocities. Results of the study with steel spheres and deforming lead pellets are described. A mechanism by which high-speed rain erosion may take place on 2S aluminum and on other soft metals with similar properties is advanced. The understanding of this specific mechanism is helpful in that it supplies evidence for the development of the basic mechanism of rain erosion of materials in general including plastic materials which are of particular interest to the aircraft industry.

WADC TR 53-192 Pt 8

May 1956

SUBJECT: MECHANISM OF RAIN EROSION Part 8 On the Breakup of a Waterdrop in the Zone Behind a Detached Shock Wave  
INVESTIGATOR: Olive G. Engle  
CONTRACT: AF 33(616)-53-9  
CONTRACTOR: National Bureau of Standards

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**ABSTRACT:** The change of pressure, density, and velocity through the shock wave caused by an object moving at supersonic velocity in still air, and the change of pressure, density, and velocity through the zone of separation between the shock wave and the object are calculated. The mechanism of water-drop breakup is discussed and the critical diameter of a waterdrop that should be able to survive in the conditions in the zone behind a detached shock is found.

WADC TR 55-319

May 1956

**SUBJECT:** WEATHERING OF GLASS-FABRIC-BASE PLASTIC LAMINATES  
**INVESTIGATOR:** Fred Werren  
B. G. Heebink  
**CONTRACT:** DO (33-616)-53-20  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Eleven different glass-fabric-base plastic laminates, made with 9 different laminating resins, were subjected to outdoor weathering at 5 sites having entirely different weather conditions. After completion of the exposure cycles, the laminated panels were tested in tension, compression, and flexure at the U. S. Forest Products Laboratory. Data on the effect of exposure on the mechanical properties and the appearance of the laminates after exposure periods of 3 months and 12 months are presented in this report.

Another series of panels is still undergoing exposure at all sites, and this series will be tested after 3 years' exposure. Upon completion of tests of these panels, all data will be compiled in a final report.

Data obtained from the tests made to date show that the effect of outdoor exposure varies with the type of laminate and condition of exposure. The laminate materials showed no severe reductions of strength after outdoor exposure for one year. Most of the materials showed average reductions from 5 to 10 percent. However, one type of material, that being the heat-resistant polyester resin laminates, showed reduction in strength averaging about 20 percent.

WADC TR 55-342

March 1956

**SUBJECT:** INVESTIGATION OF HEAT RESISTANT POLYESTER LAMINATES  
**INVESTIGATOR:** Norman E. Wahl  
Norman M. Preston  
**CONTRACT:** AF 33(616)-2515  
**CONTRACTOR:** Cornell Aeronautical Laboratory, Inc.  
**ABSTRACT:** Various methods of eliminating the cracking of triallyl-cyanurate copolymer resins reinforced with 181-301 glass cloth were investigated.

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The use of .010" asbestos surface overlay on glass cloth laminates used with commercially available triallylcyanurate resins was found to be the most practical approach to prevent cracking in production applications.

Several new triallylcyanurate copolymers that exhibit reduced water absorption and heat loss were developed. Data on two resins which eliminated the cracking are described.

WADC TR 56-206  
ASTIA Document No. AD 97314  
OTS Release

September 1956

SUBJECT: MECHANICAL PROPERTIES OF POLYESTER LAMINATES REINFORCED WITH HIGH MODULUS GLASS FABRIC  
INVESTIGATOR: Fred Werren  
CONTRACT: DO 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Comparative strength tests were made of three polyester laminates fabricated at the U. S. Forest Products Laboratory. Two of the laminates were reinforced with H. M. 18 glass fabric, a fabric designed to produce laminates with a high modulus. One laminate had a resin content of 41.9 percent and the other of 34.8 percent. The third laminate was a typical laminate reinforced with 181 glass fabric, and it had a resin content of 36.7 percent. Tension, compression, and flexure tests were made of each laminate, and the strength and elastic properties are presented in this report. The average mechanical properties of the 181 laminate were generally as good as or higher than those of the 2 H. M. 18 laminates.

Some comparative strength data on an H. M. 18 polyester laminate fabricated and tested by the Cincinnati Testing and Research Laboratories are also included in this report. The resin content of this laminate was only 29.1 percent, and the mechanical properties were higher than those of the laminates with greater resin content fabricated at the Forest Products Laboratory.

The results of this study show that the mechanical properties of an H. M. 18 laminate at a resin content of about 36 percent would probably be about the same as or lower than those of a 181 laminate at a comparable resin content. The data indicate, however, that a reduction in resin content may appreciably increase the strength and elastic properties of an H. M. 18 laminate. This relationship is also true for a 181 laminate; however, there is some indication that the increase in mechanical strength with decrease in resin content in a 181 laminate is not as great as for a H. M. 18 laminate.

April 1957

SUBJECT: SHORT-TIME ELEVATED-TEMPERATURE TENSILE AND COMPRESSION  
PROPERTIES OF GLASS-FABRIC-PLASTIC LAMINATES  
INVESTIGATOR: John A. Van Echo  
Gale R. Remely  
Ward F. Simmons  
CONTRACT: AF 33(616)-3215  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Basic property data on various heat-resistant reinforced and transparent plastic materials have been obtained for the benefit of airframe designers. Materials discussed in this report include three reinforced plastics made with typical heat-resistant types of silicone, phenolic, and polyester triallyl cyanurate resins..

Based on test results contained herein, which consists of short time static tensile and compression data after exposure to various temperatures and conditioning periods, it is shown that, generally the phenolic resin laminate possesses the highest strength after short time exposures at temperatures as high as 500° F. The silicone resin material exhibits a superior strength at the higher temperatures and long exposures. For some intermediate temperatures and exposure times, the triallyl cyanurate-polyester resin laminate has the superior comparative strength. Thus, each of the three test materials possesses superior strength properties at some of the test conditions with no one laminate being superior at all conditions.

The results presented are considered merely as an indication or trend of strength properties rather than as precise design information. As a minimum, check data should be obtained on the particular lot or batch of material actually being used.

WADC TR 56-264  
ASTIA Document No. AD 110459  
OTS Release

October 1956

SUBJECT: EVALUATION OF LOW-DIELECTRIC GLASS FABRIC  
INVESTIGATOR: Fred Werren  
- B. G. Heebink  
CONTRACT: DO 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Glass-fabric-base plastic laminates, reinforced with fabric made of low-dielectric glass fibers and standard "E" glass fibers, were evaluated on the basis of mechanical and electrical properties. Six different

laminating resins were used. Comparative data obtained from these tests showed that laminates made with low-dielectric fabric were superior in electrical properties but inferior in mechanical properties to comparable laminates made with the standard "E" glass fabric.

WADC TR 56-296  
ASTIA Document No. AD 97254  
OTS Release

September 1956

SUBJECT: NUCLEAR RADIATION OF REINFORCED PLASTIC RADOME MATERIALS  
INVESTIGATOR: Robert C. Tomashot  
Douglas G. Harvey, 1/Lt

ABSTRACT: Nine different reinforced plastic laminate materials, each made with a different laminating resin, and one alkyd-isocyanate foam core-glass fabric faced sandwich material were subjected to integrated gamma radiation dosages up to  $10^9$  roentgens. After completion of the radiation exposures, the materials were tested to determine the flexural, tensile, and compression strengths under both standard and wet conditions. The mechanical properties of the heat resistant plastic laminates were also determined at elevated temperature.

Dielectric constant and loss tangent measurements were conducted on irradiated samples to determine the effect of radiation on the electrical properties of these materials.

Data obtained from these tests show that the mechanical properties were not significantly affected except for one epoxy type resin laminate. None of the materials showed any significant change in electrical properties due to radiation.

WADC TR 56-522  
ASTIA Document No. AD 118098  
OTS Release

March 1957

SUBJECT: EFFECT OF THICKNESS ON STRENGTH OF EPOXY AND PHENOLIC LAMINATES  
REINFORCED WITH GLASS FABRIC  
INVESTIGATOR: Kenneth H. Boller  
CONTRACT: DO 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory

ABSTRACT: Results of tensile and compressive tests on laminates made with 3 types of resin and 2 types of reinforcement in thicknesses ranging from 1/100 inch to 1/8 inch showed that the stress at failure decreased with decreasing thickness. The magnitude of the decrease varies with the material. The decrease for 0.030 inch material, about the thinnest used in aircraft, may be as great as 12 percent of the strength of 1/4 inch thick material.

April 1957

SUBJECT: ROOM- AND ELEVATED-TEMPERATURE PROPERTIES OF NA-91LD  
PHENOLIC RESIN LAMINATE  
INVESTIGATOR: John A. VanEcho  
James W. Garbrant  
Ward F. Simmons  
CONTRACT: AF 33(616)-3215  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report contains engineering data for a glass fiber laminate fabricated under low (vacuum-bag) pressures from 91LD phenolic resin and 181-Volan A glass cloth. The material is identified as NA-91LD in this report. Some comparative data are included for another phenolic resin laminate (CTL-91LD) made under high mold pressures.

The property data reported include short-time tensile and compression, creep and creep-rupture in tension and compression, flexure, and deterioration. Temperatures employed range from 80 to 1000 F with prior exposures at these temperatures ranging from zero holding time to 1000 hours.

This phenolic resin laminate shows relatively good elevated- and room-temperature strength properties. Results have been fairly reproducible and consistent among the four panels tested. The flexural strength and deterioration properties of the vacuum-bag molded laminate are very similar to those of the press-molded phenolic. The creep and creep-rupture and short-time tensile strengths of the vacuum-bag laminate appear, generally, to be superior to the press-molded material. The reverse is true with regard to the short-time compression strength and elastic modulus properties.

Improvements in the finish on the glass fabric, curing techniques, and resin processing methods can considerably increase the strength properties that can be obtained with high pressure CTL-91LD laminates. However, the test data reported represent the values obtained from laminates fabricated under specific conditions and are useful in showing the properties available by this method of fabrication.

April 1957

SUBJECT: ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF EPON-  
PLYOPHEN PLASTIC-GLASS FABRIC LAMINATE  
INVESTIGATOR: Gilbert M. Gynn  
John A. VanEcho  
Ward F. Simmons

CONTRACT: AF 33(616)-3215  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Basic design data on heat-resistant laminated and transparent plastics have been obtained for the benefit of airframe designers. The material discussed in this report is a glass fabric reinforced plastic made with a particular epoxy-phenolic resin.

The test results contained in this report were obtained from creep and creep-rupture, flexural, deterioration, and short-time tensile and compression tests. These results show that after exposures ranging from 0 to 1000 hours at test temperatures of 80 to 1000°F the material displays a variety of properties. In general, the ultimate strengths decrease with increases in temperature and exposure time. This trend of decreasing strength with increasing temperature was not of a constant rate, however, for the various types of tests.

At the lower temperatures, the compression strength of the material was found to be greater than the tension strength but lesser than the flexural strength. At higher temperatures, however, the tensile strength is greater than either the flexural or compression strengths.

The results presented in this report are not to be considered precise design information, but rather a general indication of strength properties.

#### POLYMERS AND SYNTHESIS STUDIES

WADC TR 54-599 Pt 2  
OTS Release

May 1956

SUBJECT: ELASTOMERIC POLYPHOSPHATES  
INVESTIGATOR: R. A. Hubbard II  
U. P. Strauss  
CONTRACT: AF 33(616)-2059  
CONTRACTOR: Rutgers University  
ABSTRACT: The purpose of the work was to prepare an elastomer whose back-bond is the polymetaphosphate chain. The part with which this report is concerned was the cross-linking of polyphosphate chains. We attempted to prepare the possible cross-linking agent neopentane tetrakisphosphonic acid and its derivatives. A mixture of acids was prepared by the Arbuzov rearrangement from pentaerythrityl bromide which appears to be primarily the tri-phosphonic acid. Attempts to prepare a cross-linked polyphosphate from this mixture by fusion of the phosphonic acid with  $\text{KH}_2\text{PO}_4$  at 320°C were unsuccessful, due to the thermal instability of the neopentane carbon structure.

The molecular weights of several samples of potassium polyphosphate were determined viscometrically in order to obtain an idea of the degree of polymerization of the samples used in the cross-linking studies. It was found that the molecular weight of crystallized  $(KPO_3)_x$  can reach  $3 \times 10^6$ .

WADC TR 55-26 Pt III  
OTS Release

May 1956

SUBJECT: RESEARCH ON BORON POLYMERS  
INVESTIGATOR: William L. Ruigh Michael Sedlak  
Frank C. Gunderloy, Jr. P. A. Van Der Meulen  
CONTRACT: AF 33(616)-2057  
CONTRACTOR: Rutgers University  
ABSTRACT: A new, simple, and efficient synthesis of boron substituted borazoles has been developed. Butylboron dichloride reacts with ammonia to give a high yield of B-tributylborazole. This synthesis will be broadened by the employment of substituted amines. Some of the resulting B-N substituted borazoles may serve as the basis of thermally and hydrolytically stable semi-inorganic polymers and the liquid borazoles will be evaluated as lubricants and hydraulic fluids.

The preparation of benzeneboronic acid, tri-n-butylborine, butylboron dichloride and phenylboron dichloride as intermediates for our new borazole synthesis has been studied.

A new catalytic recirculating apparatus for preparing phenylboron dichloride from benzene and boron trichloride by Pace's method is described.

WADC TR 55-26 Pt IV  
ASTIA Document No. AD 110403  
OTS Release

September 1956

SUBJECT: RESEARCH ON BORON POLYMERS  
INVESTIGATOR: William L. Ruigh Nathan G. Steinberg  
Arthur D. Clin Peter A. Van Der Meulen  
CONTRACT: AF 33(616)-2057  
CONTRACTOR: Rutgers University  
ABSTRACT: Tri-B- $\beta$ -chloroborazole has been prepared from  $\beta$ -chlorovinylboron dichloride and ammonia. It is unstable and in the presence of polar solvents forms either cyclic or long chain polymers.

Large samples of 100 grams or more of Tri-B-b-butylborazole, Hexabutylborazole, and Tri-N-Methyl-tri-B-n-butylborazole were prepared for evaluation.

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May 1956

SUBJECT: DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS  
INVESTIGATOR: Paul Tarrant  
George W. Dyckes  
CONTRACT: AF 33(600)-26593  
CONTRACTOR: Peninsular ChemResearch, Inc.  
ABSTRACT: A study has been made of the effect of various initiators or catalysts on the addition reaction involving a single olefin-silane system. The use of platinum supported on charcoal gives highest yields and allows shorter reaction times and lower temperatures for this reaction.

The preparation of the monomers,  $\text{CF}_3\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$ ,  $\text{CH}_3\text{CH}(\text{CF}_3)\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$ ,  $\text{CF}_3\text{CF}_2\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$ , and  $\text{CF}_3\text{CF}_2\text{CF}_2\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$  has been carried out on a larger scale and appreciable quantities have been made for polymerization studies.

Compounds resulting from the addition of  $\text{CF}_2\text{Br}_2$  and  $\text{CF}_2\text{-Br}$   $\text{CFC1Br}$  to vinyltrimethylsilane have been made to undergo reactions involving loss of halogen. The properties of the new unsaturated compounds have been determined.

WADC TR 55-221 Pt 2

May 1956

SUBJECT: INVESTIGATION OF CONDENSATION TYPE ELASTOMERS  
INVESTIGATOR: George C. Schweiker  
Russell R. White  
Rudolph N. Deleo  
CONTRACT: AF 33(616)-2421  
CONTRACTOR: Hooker Electrochemical Company  
ABSTRACT: The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. High thermal stability ( $350^\circ\text{F}$  or higher); resistance to aromatic fuels, synthetic ester-base oils and hydraulic fluids; resistance to ozone and to weathering oxidation effects; resistance to acids, bases, and salts; resistance to abrasion; and satisfactory performance at  $-65^\circ\text{F}$  or lower are major requirements for such an elastomer.

To this end, a number of linear polyesters have been prepared from fluorine-containing diols and various dicarboxylic acid chlorides, and their properties determined. Hexafluoropentylene adipate (made from adipyl chloride and 2,2,3,3,4,4-hexafluoropentanediol) is a rubber-like gum when of sufficiently high molecular weight, and this polyester has been cross-linked to evaluate the properties of the elastomer in the light of Air Force requirements.

When reinforced with carbon black, the polymer exhibits very good physical properties (tensile strengths above 2600 psi with corresponding elongations above 300%, at the present stage of development). Preliminary evaluations show that this elastomer is thermally stable (serviceable for extended periods to 350°F or higher in air); very resistant to aromatic fuels, synthetic ester-base oils and hydraulic fluids, and acid and salt solutions at room temperature; and not brittle at -65°F or lower. It is not long resistant at the present stage of development to diester fluids at 350°F, however, or to base solutions at room temperature.

It is believed that this rubber is the first example of a fuel resistant, thermally stable elastomer which does not become brittle at low temperatures.

WADC TR 55-374

April 1954

SUBJECT: POLYMERS AND COPOLYMERS OF N-1,1-DIHYDROFLUOROALKYL ACRYLAMIDES  
INVESTIGATOR: Fred W. Knobloch  
ABSTRACT: A series of six new fluorinated acrylamide monomers have been studied. These included 1,1-dihydrotrifluoroethyl acrylamide, 1,1-dihydroheptafluorobutyl acrylamide and the N-methyl, N-n-butyl, and N-isobutyl derivatives of the latter.

Homopolymerization of the fluoroacrylamide monomers readily proceeded both in bulk and in solution with benzoyl peroxide. Liquid monomers polymerized in emulsion with persulfate initiators. All homopolymers were thermoplastics which could be cast to yield clear transparent films. The electrical, thermal, and dilute solution behavior of some of these materials has been studied.

Copolymerization proceeded with a number of co-monomers which included alkyl acrylates, fluoroalkyl acrylates, vinyl ethers, and several dienes. Essential parameters such as monomer reactivity ratios, intrinsic viscosities and empirical slope constants  $k'$  were evaluated for selected copolymers.

WADC TR 55-453

May 1956

SUBJECT: DEVELOPMENT OF INORGANIC POLYMER SYSTEMS  
INVESTIGATOR: Carlin F. Gibbs George Shkapenko  
Harold Tucker John C. Park  
CONTRACT: AF 33(616)-2744  
CONTRACTOR: The B. F. Goodrich Company

WADC TR 53-373 Sup 4

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**ABSTRACT:** Following an initial investigation of modification of phosphonitrile chloride, which was then deemphasized to avoid contractual conflicts, a study of semi-inorganic polymers based upon an aluminum-oxygen-silicon system was begun. The basic reasons for studying such a system were founded upon the known thermal stability of aluminum silicates found in nature.

The most stable difunctional aluminum monomers found were quadricovalent chelates such as mono (1,3-diphenyl-1,3-propanedione) aluminum diisopropoxide which melted at 324°C. without decomposition.

Reaction of the difunctional aluminum monomers with difunctional silicon monomers has not yet resulted in high molecular weight materials.

WADC TR 56-82

May 1956

SUBJECT: PRINCIPLES OF INORGANIC POLYMERIZATION  
INVESTIGATOR: Anton B. Burg Walter Mahler  
Peter J. Slota, Jr. Gordon L. Juvinall  
CONTRACT: AF 33(616)-2743

CONTRACTOR: University of Southern California  
ABSTRACT: The object of this research is to discover new patterns of chemical bonding such as might be applicable to the invention of high polymers having unusual thermal stability. It is believed that a considerable number of structural arrangements of nonmetallic elements can be stronger than carbon chains, and more resistant to thermal degradation. Leads in this direction are found as phosphorus-boron bonding is subjected to further study, for the amplification of which the attachment of nonlabile electronegative groups to boron is regarded as useful. The P-N polymer system also seems promising, and methods are sought for placing new and nonlabile substituents on P in (PN)<sub>n</sub> rings. Eventually such substituents should include cross-linking units, and this is one reason for studying a newly-discovered variety of fluorocarbon-phosphorus compounds wherein P-C-P rings and (-C-P-)<sub>n</sub> chains are indicated.

As indicated in the summary, the work of the first year of this contract has developed a number of promising lines of endeavor, most of which cannot be fully understood and evaluated except through more detailed studies. Hence their utility is a matter for the future to decide.

WADC TR 56-82 Pt II  
ASTIA Document No. AD 118301  
OTS Release

May 1957

**SUBJECT: PRINCIPLES OF INORGANIC POLYMERIZATION**

INVESTIGATOR: Anton B. Burg  
Gottfried Brendel  
Aimery P. Caron  
Gordon L. Juvinall  
Walter Mahler  
Kurt Modritzner  
Peter J. Slota, Jr.

CONTRACT: AF 33(616)-2743

CONTRACTOR: University of Southern California

ABSTRACT: This project continues to seek and to find new patterns of chemical bonding and to consider their possible usefulness in relation to the invention of new polymers having unusual thermal stability. Some of our amino-phosphino-boron resins are stable at temperatures approaching 500°C., and the limit is not known; however the problem of their poor mechanical properties remains. The phosphinoborine bonding principle withstands a great increase in the electronegativity of substituents on phosphorus. The stability of P-P bonding seems much enhanced by  $\text{CF}_3$  groups on P, so that the new compounds  $(\text{PCF}_3)_4$  and  $(\text{PCF}_3)_5$  are decidedly stable. The previous tentative interpretations of the by-products of the fluorocarbon-phosphorus iodide reactions with mercury are reconsidered in the light of new facts. Progress toward more electronegative substituents on boron is reported, and our recently initiated work on phosphino-aluminum compounds has produced some interesting materials. Nitrilophosphoric chloride is not depolymerized by pyridine, but it reacts with triethylamine in an interesting manner.

WADC TR 56-90

May 1956

SUBJECT: INVESTIGATION OF INORGANIC AND SEMI-ORGANIC POLYMERS

INVESTIGATOR: Samuel Witz

CONTRACT: AF 33(616)-2739

CONTRACTOR: Aerojet-General Corporation

ABSTRACT: A. The present investigation sought to determine whether a polymer containing a Si-O-P or a B-O-P linkage and possessing high-temperature stability and resistance to hydrocarbon fluids could be synthesized.

B. Several new polymeric materials containing a Si-O-P skeleton were synthesized. Most of these solids were low-melting, tacky, brittle resins of low molecular weight and were sensitive to moisture.

C. Fundamental information about the behavior of the Si-O-P linkage toward heat and hydrolysis was obtained through a study of some newly synthesized alkylsilyl phosphates  $[\text{R}_2\text{SiO}]_2\text{PO}$  and phosphonates  $[\text{R}_2\text{SiO}]_2\text{P}(\text{O})\text{R}'$ . The hydrolytic stability of the high-boiling phosphates was found to increase with increasing chain length of the silicon substituent. Both the phosphate and phosphonates undergo some decomposition on pyrolysis at 300°C (in vacuo).

D. In a study designed to test the effectiveness of dialkyl alkanephosphonates as heat-stabilizing additives, it was found that the addition

of a small percentage (3.5%) of dibutyl butanephosphonate to a commercially available silicone resin improved its thermal properties.

E. Condensation of benzeneboronic and cyclohexanephosphonic acids produced an organoboron phosphate, melting above 300°C, possessing a limited solubility in organic solvents, but soluble in and hydrolyzed by water.

WADC TR 56-94  
OTS Release

May 1956

SUBJECT: THE PREPARATION AND PROPERTIES OF SOME NEW FLUORINE-CONTAINING 1,2-EPOXIDES  
INVESTIGATOR: D. A. Rausch, 1/Lt  
A. M. Lovelace, 1/Lt  
ABSTRACT: The interest in fluorine-containing polyethers for possible application as thermally stable elastomers prompted research on the preparation of new epoxide monomers.

Six new epoxides were prepared and characterized. The utilization of these monomers in new polymer systems is being investigated.

WADC TR 56-201  
OTS Release

May 1956

SUBJECT: RESEARCH ON THE SYNTHESIS OF POLAR SILANE MONOMERS  
INVESTIGATOR: Robert M. Silverstein  
Leon Goodman  
Allen Benitez  
CONTRACT: AF 33(616)-2998  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: This report summarizes the work done between May 1955 and May 1956 on the synthesis of polymerizable polar silane monomers and the copolymerization of certain of these with dimethylsilicone.

The principal method used for the monomer synthesis was the addition of the Si-H linkage to vinyl and allyl monomers. Monomers containing halogens were prepared by the Grignard reaction, by Diel-Alder addition of hexachlorocyclopentadiene to unsaturated silanes, and by the addition of poly-halogenated molecules to unsaturated silanes.

Hydrolysates of certain of the polarsilane monomers strongly inhibited polymerization when mixed in 5 mole percent amounts with the hydrolysate of dimethyldichlorosilane. The polymerization could be forced but the products were then insoluble in benzene.

WADC TR 53-373 Sup 4

October 1956

SUBJECT: COORDINATION POLYMERS  
INVESTIGATOR: W. Conard Fernelius  
CONTRACT: AF 33(616)-2742  
CONTRACTOR: The Pennsylvania State University  
ABSTRACT: The purpose of the work is to provide research and development on organic metal-coordination polymers, which exhibit exceptional thermal stability.

The concept of producing polymers through coordination is presented, pertinent literature reviewed, and various possibilities of developing such polymers considered.

Chelate polymers of bis ( $\beta$ -diketones) were prepared under varying conditions and their physical properties determined. Molecular weights range up to 6000.

Attempts to form polymers from bis(Schiff bases) and to produce a truly inorganic polymer by the coordination of polyfunctional acids (anions) to complex cations have not yet been successful.

November 1956

SUBJECT: PREPARATION OF FLUORINE-CONTAINING COMPOUNDS  
INVESTIGATOR: H. C. Brown J. A. Wethington, Jr.  
R. D. Dresdner J. A. Young  
CONTRACT: NONR 580(03)NR 356-333  
CONTRACTOR: University of Florida  
ABSTRACT: Part of the objective of this program is the preparation or attempted preparation of new fluorine-containing chemical species by the Simon's electrochemical process. The results of numerous operations in a variety of cells are summarized. Studies have been made with some of the resulting materials in order to find unique reactions of these materials and thereby new compounds exhibiting the chemical and thermal stability associated with fluorocarbons.

The preparation, pyrolysis or thermal reactions of certain simple fluorocarbon sulfides with fluorocarbon olefins are described. The sulphides are  $\text{CF}_3\text{SF}_5$ ,  $\text{C}_2\text{F}_5\text{SF}$ ,  $(\text{CF}_3)_2\text{SF}_4$  and  $(\text{C}_2\text{F}_5)_2\text{SF}_4$ . The olefins involved were  $\text{CF}_3\text{CF}=\text{CF}_2$ , and  $\text{CF}_3\text{N}=\text{CF}_2$ .

The synthesis and properties of fluorocarbon derivatives having hetero atoms such as oxygen or nitrogen in the principal carbon chain of the molecule have been studied. It has been shown that a perfluoro compound having an alternating chain C-O-C-N-C cannot be synthesized directly from the organic analogue. When prepared indirectly, however, such a chain is chemically and thermally stable. The compounds  $(\text{CF}_3)_2\text{NCOF}$ ,  $\text{CF}_3\text{N}=\text{CF}_2$ , and  $(\text{CF}_3)_2\text{NCF}_2\text{COOH}$ , which are the N-hetero analogues respectively of  $(\text{CF}_3)_2\text{CFCOF}$ ,  $\text{CF}_3\text{CF}=\text{CF}_2$ , and  $(\text{CF}_3)_2\text{CFCF}_2\text{COOH}$ , are discussed. The pyrolysis of a compound having C-C, C-O, and C-N bonds is described. Preparation of a new and unexplored class of compounds, the perfluoroamides, and the synthesis of a possible intermediate in the preparation of the difunctional acid  $\text{O}(\text{CF}_2\text{COOH})_2$  are mentioned.

Reaction of fluorocarbon olefins and chlorofluoroolefins has produced sulfur-containing fluorocarbon derivatives stable to aqueous alkali. Preparations and reactions of perfluoroalkyl amidines,  $\text{R}_\text{F}\text{C}(\text{:NH})\text{NH}_2$ , and perfluoroalkyl triazines,  $(\text{R}_\text{F}\text{CN})_3$  have been studied. The reaction of polytetrafluoroethylene with sulfur or selenium under pyrolytic conditions produces a fusible polymer containing no sulfur.

Efforts to use potassium and sodium vapor as a reaction medium for fluorocarbon synthesis have not been successful. Fluorine exchange between metal fluorides and fluorocarbon olefins has been studied. Exchange was generally accompanied by decomposition. It was found that the reactor packing determined the nature of the products obtained from the oxidation of  $\text{C}_3\text{F}_6$ .

WADC TR 56-326  
ASTIA Document No. AD 110712  
OTS Release

January 1957

SUBJECT: CHLOROSULFONATION OF SILANES AND RELATED REACTIONS  
INVESTIGATOR: C. G. Overberger  
F. M. Beringer  
CONTRACT: AF 33(616)-2997  
CONTRACTOR: Polytechnic Institute of Brooklyn  
ABSTRACT: The aim of this work was the synthesis of polar organo-silicon monomers capable of polymerization to synthetic rubber. The polar groups would be expected to decrease the solubility of the silicone rubber in organic solvents as well as increasing the transition temperature.

The synthesis of a suitable polar organosilicon monomer has not yet been effected. Our results, some of which represent progress toward the desired goal, are now summarized.

(1) The direct photochemical chlorosulfonation of silanes with sulfuryl chloride was attempted without success. In one case a small amount of cleavage product, methanesulfonyl chloride was formed.

(2) The direct chlorocarbonylation of tetraethylsilane with oxalyl chloride was effected.

(3) Three silanes probably containing the  $-\text{CH}_2\text{SCN}$  group have been prepared. However, attempted conversions to compounds with  $-\text{CH}_2\text{SO}_2\text{Cl}$  groups have failed.

(4) Vinyltriethoxysilane with methylmagnesium iodide has given vinylmethyldiethoxysilane and vinyltrimethylethoxysilane.

(5) Benzyl and phenyl mercaptans have been added to the above vinylsilanes to give substituted beta-thioethylsilanes. Attempts to oxidize these sulfides to sulfones have led to mixtures of materials of higher molecular weight.

(6) Hydrolysis of various silane sulfides has given mixtures of silane sulfides, as yet incompletely characterized.

While silicones with polar substituents would still seem to be desirable and useful materials, the synthesis of suitable monomers has not been accomplished in the present work.

WADC TR 56-399  
ASTIA Document No. AD 110557  
OTS Release

November 1956

SUBJECT: POLYMER EVALUATION HANDBOOK  
INVESTIGATOR: C. H. Adams G. B. Jackson  
R. J. Bourke J. R. Taylor  
CONTRACT: AF 33(616)-3034  
CONTRACTOR: Monsanto Chemical Company  
ABSTRACT: The Polymer Evaluation Handbook provides a system for obtaining a maximum amount of useful information on small quantities of experimental polymers. It also provides background information on principles of evaluation, properties and measurements and their significance, the nature and behavior of polymers and physical characteristics of plastics in relation to each other and to other classes of materials

The Handbook describes a screening evaluation scheme for effectively and efficiently defining the physical and chemical behavior of small



quantities of experimental polymeric materials. The soundness of the evaluation scheme was tested in a pilot laboratory. Examples of the results of these tests are given.

Interpretation concepts are discussed which pertain to the appraisal of evaluation information and data. It is noted that the effectiveness of screening evaluations will depend to a large extent on two major factors: (1) the scientific knowledge, experience, judgment, and perception of the person conducting the evaluation, and (2) a reasonable balance between research laboratory efforts on synthesis and evaluation work.

WADC TR 56-492  
ASTIA Document No. AD 118158  
OTS Release

March 1957

SUBJECT: THE PREPARATION AND POLYMERIZATION OF PERFLUOROALKYL  
PROPENYL KETONES  
INVESTIGATOR: D. A. Rausch, 1/Lt  
L. E. Coleman, 1/Lt  
A. M. Lovelace  
ABSTRACT: A study of the preparation of  $\alpha, \beta$  unsaturated ketones containing fluorine has been undertaken in this laboratory for use as monomers in the preparation of new and useful plastic and elastomeric materials for high temperature applications in military aircraft.

The perfluoroalkyl propenyl ketones were found to copolymerize with a variety of vinyl monomers such as acrylonitrile, ethyl acrylate, styrene and vinyl acetate to give products ranging from powders to elastomers.

WADC TR 56-493  
ASTIA Document No. AD 118344  
OTS Release

May 1957

SUBJECT: PREPARATION AND POLYMERIZATION OF SOME FLUOROALKYLMETHYL-  
CYCLOSILOXANES  
INVESTIGATOR: Eugene C. Stump, 1/Lt  
ABSTRACT: Four fluoroalkylmethyldichlorosilanes were hydrolyzed and the resulting cyclosiloxanes separated and characterized.

Attempts to homopolymerize the fluoroalkylmethylocyclosiloxanes resulted in viscous fluids.

Gum copolymers with octamethylcyclotetrasiloxane were prepared by conventional methods.

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Fluorine-containing cyclosiloxanes may also be prepared from the higher molecular weight siloxane residues of the hydrolysis reaction by passing through a hot tube at 470-520°C.

WADC TR 56-530  
ASTIA Document No. AD 110717  
OTS Release

January 1957

SUBJECT: RESEARCH ON THE SYNTHESIS OF POLAR SILANE MONOMERS

INVESTIGATOR: Robert M. Silverstein

Leon Goodman

Allen Benitez

CONTRACT: AF 33(616)-2998

CONTRACTOR: Stanford Research Institute

ABSTRACT: The prime objective of this research is to synthesize polymerizable, polar silane monomers for use as precursors or modifiers for improved silicone rubbers. A second important objective of the project is to prepare gums from these monomers, homopolymerized or copolymerized with dimethylsilicone oils, such that the copolymer can be compounded to produce suitable silicone elastomers.

The structures of the  $\text{MeSiCl}_2\text{H}$  adducts with vinyl acetate and with methyl acrylate were shown, by nuclear magnetic resonance spectra, to be  $\text{MeSiCl}_2\text{CH}_2\text{CH}_2\text{OCOCH}_3$  and  $\text{MeSiCl}_2\overset{\text{CH}_3}{\text{C}}'\text{H}-\text{COOCH}_3$ , respectively. The latter compound represents the first reported example of non-terminal addition of Si-H compounds to vinyl double bonds.

Samples of dimethylsilicone copolymers containing the vinyl acetate adduct, the allyl acetate adduct, the vinyl ethyl ether adduct, and the allyl ethyl ether adduct were prepared. All of these were cross-linked and were unsuitable elastomer materials.

The reactions of the strong acids and strong bases used as polymerization catalysts, with the hydrolysates of the silane adducts, were shown to lead to C-Si bond cleavage in a number of cases. This cleavage provides a plausible explanation of the cross-linking that results when the adducts are used in copolymerization.

The use of  $\beta$ - and  $\gamma$ - radiation sources led, in preliminary studies, to surface grafts of  $\beta$ -cyanoethyl acrylate onto a commercial silicone elastomer which gave an improvement in resistance to swelling caused by 70:30 isooctane:toluene.

April 1957

SUBJECT: RESEARCH ON HIGH TEMPERATURE LAMINATING RESINS BASED ON  
MALEIMIDE AND ITS DERIVATIVES  
INVESTIGATOR: Roger W. Amidon  
Charles H. Alexander  
Frank B. Root  
CONTRACT: AF 33(616)-3606  
CONTRACTOR: Naugatuck Chemical Company  
ABSTRACT: The objective of this contract was the synthesis of laminat-  
ing resins for service in the range of 600° to 700°F, based primarily on  
maleimide and on derivatives thereof.

Test laminates were prepared of various blends of maleimide with standard heat-resistant alkyds. Laminates from one such blend had a raw flexural strength of 67,100 psi, as compared to 37,600 and 42,100 psi for VIBRIN 135 and X-1068 laminates respectively. After 24 hours aging at 500°F, it retained a flexural strength of 61,200 psi as compared to 35,400 and 36,000 psi, and after 192 hours at 500°F, 34,500 psi as compared to 19,300 and 18,200 psi, measured at 500°F.

Copolymers of maleimide and methylol maleimide with several vinyl type monomers were prepared and studied as possible heat resistant resin components. None of the materials but one showed any promise of desirable properties, and further investigation along this line is not considered profitable.

Synthesis of three monomeric unsaturated compounds containing the maleimide nucleus was attempted. It appears the synthesis of N-Allyloxy-methyl maleimidewas successful, however, complete analysis of the reaction product is not yet available.

May 1957

SUBJECT: DEVELOPMENT OF THERMALLY STABLE SILICON CONTAINING RESINS  
INVESTIGATOR: L. W. Breed  
Fred Baiocchi  
Howard W. Christie  
CONTRACT: AF 33(616)-3675  
CONTRACTOR: Midwest Research Institute  
ABSTRACT: The purpose of this program is the development of thermally  
stable silicon containing resins, and the parts of that program already completed  
are described in full. This program includes the synthesis of silane monomers

which contain two silicon atoms connected by arylene groups and have sufficient functionality to form a thermosetting polymer when hydrolyzed and cured. It also includes finding methods of hydrolyzing and polymerizing these monomers in which the maximum use is made of their functionality.

Possible methods for synthesizing the monomers have been investigated. Those methods which were found suitable for the preparation of quantities of one pound or more were used to prepare the following compounds: 1,4-bis(methyldiethoxysilyl)benzene, 4,4'-bis(methyldiethoxysilyl)phenyl-ether, and phenyldichlorosilyl-trichlorosilylbenzene. Also, the description of a statistically designed series of experiments to study the variables effective in the formation of laminates from silicone resins and glass fiber fabric is included.

### RUBBER

WADC TR 52-197 Pt 5  
OTS Release

April 1956

SUBJECT: SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS  
INVESTIGATOR: Frank A. Bovey  
CONTRACT: AF 33(038)-515  
CONTRACTOR: Minnesota Mining and Manufacturing Company  
ABSTRACT: Fluorine containing elastomers, such as FBA (1,1-dihydroper-fluorobutyl acrylate), are being prepared for possible use in fuels, lubricants, and hydraulic fluids over the widest possible temperature range (<0°F to <350°F).

Fluorinated diene-vinyl ether type copolymers continue to present difficulties in preparation. Electron irradiation has been an effective means of vulcanization.

Copolymers of 1,1,2-trifluorobutadiene with FBA have good tensile strength, solvent resistance, and low temperature behavior but lack resistance to heat and ozone.

Certain antioxidants increase the resistance of FBA polymers to dry heat. Thioamines give promise as high temperature stabilizers for poly-FBA.

Traces of co-polymerized acrylic acid are important for optimum cured properties of poly-FBA.

Butyl carbitol formal is one effective non-fluorinated plasticizer for poly-FBA but is subject to solvent extraction. Only polymeric materials resist such solvent extraction. Marked plasticization can be accomplished with silicone polymers, but swelling is excessive. Blends of Teflon and poly-FBA appear promising in strength and solvent resistance, but poor in uniformity and high temperature properties.

The greatest improvement in low temperature flexibility, without sacrifice of other desirable properties as compared to poly-FBA, is achieved by incorporating ether oxygen links into the alcohol side chain of the acrylate structure.

WADC TR 52-197 Pt 6  
ASTIA Document No. AD 97330  
OTS Release

September 1956

SUBJECT: SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS  
INVESTIGATOR: Carl A. Dahlquist  
CONTRACT: AF 33(038)-515  
CONTRACTOR: Minnesota Mining and Manufacturing Company  
ABSTRACT: This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Certain fluorine-containing alkoxy acrylate rubbers are superior to poly-FBA in serviceability at low temperature, and equal to poly-FBA in resistance to fuels and hydraulic fluids. The alkoxy acrylate rubbers which have been investigated are poly-FMFPA (poly-3 perfluoro-methoxy-1,1-dihydroperfluoropropyl acrylate), and poly-FEFPA (poly-3-perfluoroethoxy, -1,1-dihydroperfluoropropyl acrylate), and poly-H-FEFPA (poly-3( $\alpha$ -hydroperfluoroethoxy)-1,1-dihydroperfluoropropyl acrylate). Poly-H-FEFPA is more sensitive to ketone and ester solvents than the other rubbers mentioned above.

A copolymer of perfluorobutadiene and vinyl 1,1-dihydroperfluorobutyl ether continues to be of interest for its resistance to hot air aging. Good vulcanizates have not been obtained because the copolymer crosslinks badly during polymerization.

Process changes which facilitate production of poly-FBA have been approved.

The amine curing recipe for poly-FBA is still the preferred recipe for best mechanical properties. Attempts to develop latent curatives for compounds with better bin life than the TETA recipe have been unsuccessful.

Vulcanizates of blends of poly-FBA and Linde silicone rubber W-96 containing 50% or less poly-FBA exclusive of fillers and curatives have excellent low temperature flexibility, low volume swell in phosphate ester hydraulic fluids, and good resistance to hot air aging.

Surface esterified silica (Valron) has given excellent reinforcement of poly-FBA, but is no longer commercially available. Valron filled stock is far less permeable to Freon 22 than black filled stock.

WADC TR 55-58  
ASTIA Document No. AD 97234  
OTS Release

August 1956

SUBJECT: A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS

INVESTIGATOR: D. L. Loughborough J. R. Beatty  
A. E. Juve J. W. Born

CONTRACT: AF 33(616)-2308

CONTRACTOR: The B. F. Goodrich Company

ABSTRACT: Comprehensive study of the effects of nuclear radiation upon elastomeric compounds and compounding ingredients was the main purpose of the development. The work was done at the B. F. Goodrich Company Research Center. Two hundred nineteen compounds were selected for study. One hundred ninety were irradiated and tested. Stress-strain and stress relaxation measurements were made, along with special analyses of irradiation products. The investigations exhibit three principal results: (1) a catalog of stress-strain data for many elastomeric formulations has been compiled, (2) a group of inhibitors of radiation deterioration in rubber has been discovered, and (3) evidence has been gathered that the effect of Cobalt 60 gamma irradiation on rubber is different in air than in high vacuum. The rubber compounds which are most resistant to deterioration are cited by recipe.

WADC TR 55-58 Pt II  
ASTIA Document No. AD 97336  
OTS Release

September 1956

SUBJECT: A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS

INVESTIGATOR: John W. Born

CONTRACT: AF 33(616)-2308  
CONTRACTOR: The B. F. Goodrich  
ABSTRACT: Continuous stress relaxation measurements in air detail the effects of temperatures ranging from 25° through 80°C. on radiation damage to rubber compounds. The order of decreasing resistance to damage is natural rubber, GR-S Neoprene GN, and Hycar 1002 for gum rubber compounds and GR-S, Hycar 1002, natural rubber, and Neoprene GN among the black compounds. Graphs illustrate these results.

Stress relaxation measurements, volume swell measurements, infrared absorption analyses, and mass spectral analyses provided fundamental information about radiation damage. They showed further that heat and radiation damage differ significantly.

The Anti-Rad screening program and the study of the effect of radiation upon dynamic properties of rubber have reached the physical testing stage. Compounds containing 91 specially-selected potential Anti-Rads have received optimum cures, and samples have undergone gamma irradiation. The purpose of the screening is to provide criteria for the synthesis of specific new Anti-Rads.

WADC TR 55-206  
OTS Release

June 1956

SUBJECT: THE INVESTIGATION OF THE PREPARATION OF ACRYLON RUBBER  
MODIFICATIONS UTILIZING COPOLYMER AND TERPOLYMER SYSTEMS

INVESTIGATOR: B. David Halpern  
Wolf Karo

CONTRACT: AF 33(616)-2461

CONTRACTOR: Monomer-Polymer

ABSTRACT: In order to improve acrylate-acrylonitrile rubber formulations which exhibit good heat stability and fuel resistance, modifications of this system were investigated using trifluoroethyl acrylate as the major component. Copolymerization of acrylamides with trifluoro-ethyl acrylate gave elastomers with a number of favorable properties but with high brittle points. Attempts to improve the brittle points resulted in polymers with lower heat stability and higher volume swell. A study of trifluoroethyl acrylate-acrylonitrile copolymers was also made.

WADC TR 55-492  
ASTIA Document No. AD 118145  
OTS Release

March 1957

WADC TR 53-373 Sup 4

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SUBJECT: DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS  
RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES

INVESTIGATOR: Edward J. Fujiwara Wayne G. Lajines  
Theodore L. Eriksson Anthony P. Tochman  
Loren C. Smith

CONTRACT: AF 33(616)-2779

CONTRACTOR: Wyandotte Chemicals Corporation

ABSTRACT: This report describes research directed toward the development of rubber compounds resistant to various petroleum base fuels for long exposures (500 hours) at elevated temperatures (250 to 400°F.). At the beginning of the program, no apparatus was available which was capable of performing tests of tensile strength and elongation in the presence of the test fuels at the elevated temperatures. Consequently, a screening test was adapted to test candidate materials after exposure to the test fuels at elevated temperatures. This work was performed concurrently with the design of high temperature-total immersion test apparatus.

Initial screening tests on elastomer samples submitted by suppliers showed that target physical properties after exposure in test fuels for 500 hours at 400°F. could not be met. However, the initial data indicated that compounded materials based on fluorinated raw gums could possibly yield satisfactory products. Initial compounding and curing studies with polymers of Hycars 1001 and 4021 and subsequent screening tests on the resulting materials, to date, indicate that only marginal improvements can be made.

Two apparatus capable of performing tests of tensile strength and elongation on elastomer samples, in O-ring form, in the presence of test fuels at elevated temperatures, have been designed. A single sample tester (designated Type I) has been constructed. The design and necessary modification designs and the engineering and detail drawings of a multi-sample tester (designated Type II) have been completed. This tester is ready for immediate construction.

In future work on this problem initial major effort will be directed to the construction of the Type II Tester and necessary refinement work on both types of testers. Concurrently, studies on the development of rubber compositions based on fluorinated raw gums will be initiated with eventual shift of emphasis to this work. Other materials, both polymers and test liquids, will be considered as indicated and as requested by Wright Air Development Center.

WADC TR 56-155  
ASTIA Document No. AD 110633  
OTS Release

December 1956



SUBJECT: HIGH TEMPERATURE RESISTANT SEALANT MATERIALS  
INVESTIGATOR: Leonard C. Boller Wendell Olson  
John M. Snider Frank Hirose  
John H. Emigh  
CONTRACT: AF 33(616)-2767  
CONTRACTOR: Coast Pro-Seal and Manufacturing Company  
ABSTRACT: This project was undertaken by the Coast Pro-Seal and Manufacturing Company to develop fuel tank sealant compounds capable of withstanding fuel vapor temperatures of 540°F and liquid fuel temperatures of 380°F for a limited period of time.

A formula was developed for a sealant compound which was unaffected by JP-5 jet fuel under these conditions, retained flexibility and adhesion and withstood proof testing under flexing and pressure at the liquid and vapor fuel temperatures required.

A study was made of the thermal stability of the various commercially available types of butadiene-acrylonitrile polymers and combinations of them with various commercial phenolic resins. Studies were made with added antioxidants and in inert atmospheres.

Some preliminary work was started with coatings based on aqueous dispersions of polytetrafluoroethylene.

Test procedures were developed using the Parr high pressure apparatus for liquid fuel at 380°F and fuel vapor at 540°F. Procedures and apparatus were developed for proof testing coatings under conditions of flexing under pressure at these temperatures.

WADC TR 56-272  
ASTIA Document No. AD 110598  
OTS Release

November 1956

SUBJECT: DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS  
INVESTIGATOR: Frank W. Tipton  
CONTRACT: AF 33(616)-2867  
CONTRACTOR: Boeing Airplane Company  
ABSTRACT: In order to fully utilize materials for seal design, a knowledge of the relationship between the physical properties of the materials and sealing efficiency is required. This report includes a literature survey on O-rings and seal design and functional tests of O-rings prepared from rubber compounded to have various physical properties. No definite relationship between seal life and physical properties was found. The inability of the O-ring to maintain sufficient internal pressure and to withstand mechanical conditions are believed to be the two reasons for seal failure.

December 1956

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS  
INVESTIGATOR: Floyd M. Smith Leland J. Kitchen  
Thomas F. Lavery Sydney Smith  
Robert A. Hayes  
CONTRACT: AF 33(616)-3108  
CONTRACTOR: The Firestone Tire & Rubber Company  
ABSTRACT: Commercially available elastomers were compounded for high temperature performance and tested for resistance to 8-hr heat-aging up to 550°F, with the object of selecting elastomers that might prove suitable at 350-550°F for use in tires and other aircraft component parts.

Silicone rubber (Linde W-96 Silicone) and Kel-F Elastomer vulcanizates were outstanding in heat-aging resistance, requiring temperatures of 480°F and 450°F, respectively, to produce a 25% reduction in room-temperature tensile strength after 8 hrs of aging. The aging temperatures at which other elastomers retained 75% of their strengths when tested at room temperature after the 8-hr aging are as follows: Acrylon EA-5, 425°F; Hycar 4021, 395°F; Fluoro-Rubber 1F4, 385°F; Hypalon and polyurethane, 350°F; Butaprene NL (nitrile rubber), 340°F; butyl, 335°F; Neoprene Type WRT, 325°F; Thiokol St, 285°F; GR-S, 275°F; carboxy rubber, 220°F; and Hevea, 215°F.

The elevated temperatures at which some unaged elastomers retained 50% of their original room-temperature strengths are: butyl, 330°F; DC-401 Silicone, 323°F; Hevea and Acrylon EA-5, 277°F; Hycar 4021 and Thiokol ST, 255°F.

To obtain the above tensile strength information it was necessary to modify an L-6 Scott Tester, develop a suitable source of heat and temperature controls and determine the optimum techniques for obtaining reproducible results. A special permeameter was developed for determining the permeability to air and other gases at elevated temperatures, and air permeabilities were measured at 176°F and 250°F. High-temperature hardness and resilience measurements also were made.

New polymers of high thermal stability were synthesized by extending polyester chains with Epon resin, and also with 1,2,3,4-butanetetracarboxylic acid dianhydride. A special polyurethane rubber was developed which was thermally stable up to 350-400°F. This is about 50°F higher than for conventional polyurethane-type rubbers.

November 1956

SUBJECT: EFFECT OF POTENTIAL ROCKET FUELS AND OXIDIZERS ON ELASTOMERS  
AND DEVELOPMENT OF ELASTOMERIC COMPOUNDS SUITABLE FOR  
RETENTION OF THESE MATERIALS

INVESTIGATOR: C. J. Maloney  
A. S. Kidwell

CONTRACT: AF 33(616)-2962

CONTRACTOR: The Connecticut Hard Rubber Company

ABSTRACT: Kel-F Elastomer was selected on the basis of screening tests as the elastomeric material showing the best overall resistance to red fuming nitric acid and hydrocarbon rocket fuels. Blends of polyisobutylene and polyethylene, while not true elastomers, showed excellent resistance to immersion in and penetration of red fuming nitric acid; these blends also showed good resistance to n-propyl nitrate, ethylene oxide, and uns-dimethylhydrazine, but were not suitable for exposure to JP-4 fuel. Poly-FBA was fairly resistant to attack by the acid, although it swelled considerably in the acid and was relatively permeable to it. Both Kel-F Elastomer and Poly-FBA were little affected by JP-4 fuel at room temperature or at 160°F.

Kel-F Elastomer was selected for further work under the second phase of the program. Compounds cured with benzoyl peroxide or p-chlorobenzoyl peroxide far exceeded those cured with amines or isocyanates in resistance to red fuming nitric acid; optimum peroxide concentrations found were considerably lower than those normally used, reducing undesirable by-products and minimizing blistering and delamination problems in fabrication. Of the fillers tested, LM-3 silicone-coated Hi-Sil C showed the best reinforcement; others showed promise as extenders. Kel-F Elastomer was not suitable for use with n-propyl nitrate, ethylene oxide, or uns-dimethylhydrazine.

A modification of the standard H-cell permeability test showed good correlation between the water-pH values and actual metal corrosion.

RFNA with hydrogen fluoride additive showed noticeably greater effect on the various elastomers than straight RFNA.

#### SANDWICH CONSTRUCTION

September 1956

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON  
SANDWICH CONSTRUCTION FOR AIRCRAFT  
INVESTIGATOR: Donald G. Coleman  
CONTRACT: AF 33(616)-56-9  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Developments in the program of research in sandwich construction for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1956 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Nine technical reports issued during the fiscal year are abstracted.

WADC TR 55-417  
ASTIA Document No. AD 97288  
OTS Release

September 1956

SUBJECT: PERFORMANCE OF STAINLESS STEEL SANDWICH CONSTRUCTION AT  
HIGH TEMPERATURES  
INVESTIGATOR: V. C. Setterholm  
E. W. Kuenzi  
CONTRACT: DO 33(616)-53-10  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: This report presents the results of edgewise-compression and flexure tests which were conducted on stainless steel sandwich constructions at temperatures ranging from 75° to 1,200°F. Although many of the panels were corroded and poorly bonded, results of edgewise-compression tests showed that specimens from panels weighing only 2.2 pounds per square foot can sustain facing stresses as high as 230,000 pounds per square inch at 75°F. and 57,000 pounds per square inch at 1,200°F.

WADC TR 56-86  
ASTIA Document No. AD 97289  
OTS Release

September 1956

SUBJECT: EFFECT OF MOISTURE SORPTION ON WEIGHT AND DIMENSIONAL  
STABILITY OF ALKYD-ISOCYANATE FOAM CORE  
INVESTIGATOR: V. C. Setterholm  
E. W. Kuenzi  
CONTRACT: AF 33(616)-53-20  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Tests were made to determine changes in weight and dimensions of alkyd-isocyanate foam cores subjected to immersion in water and exposure to high humidity for 60 days. Cores of varying densities were investigated. The results indicated that there are differences between alkyd-isocyanate foams that have been foamed in blocks without facings and those that are foamed between two facings to make sandwich constructions. Exposure to high humidity caused increases in weight as high as 4.0 percent and dimensional increases of 0.7 percent.

WADC TR 53-373 Sup 4

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September 1956

SUBJECT: PERFORMANCE OF GLASS-FABRIC SANDWICH AND HONEYCOMB CORES  
AT ELEVATED TEMPERATURES

INVESTIGATOR: Vance C. Setterholm  
Edward W. Kuenzi

CONTRACT: DO No. 33(616)-56-9

CONTRACTOR: Forest Products Laboratory

ABSTRACT: This report presents the results of flatwise compression and flexure tests of heat-resistant glass-fabric honeycomb cores and sandwich after short and long periods of exposure to temperatures ranging from 75° to 700°F. Increases in test temperature produced reductions in shear strength and compressive strength of cores treated with phenolic, silicone, or heat-resistant polyester resin. Cores treated with phenolic resin were strongest at all temperatures. Cores treated with silicone resin suffered the least reduction in strength due to increases in test temperature, but these cores were much weaker at all temperatures than cores treated with the other resins.

October 1956

SUBJECT: PERFORMANCE OF SANDWICH WITH CORES OF FOAMED SILICONE AND  
MODIFIED POLYESTER RESINS AT ELEVATED TEMPERATURES AND AT  
HIGH HUMIDITY

INVESTIGATOR: V. C. Setterholm  
E. W. Kuenzi

CONTRACT: AF 33(616)-56-9

CONTRACTOR: Forest Products Laboratory

ABSTRACT: Compression, tension, and shear properties of plastic sandwich with foamed-in-place cores were determined at temperatures from 75° to 700°F. Cores were of Dow Corning silicone foam R-7002, with a density range of 12.6 to 17.5 pounds per cubic foot, and triallylcyanurate modified polyester resin (Laminac 4231) reacted with 2,4-toluene diisocyanate, with a density range of 11.9 to 12.2 pounds per cubic foot. Also presented are data on the weight and dimensional stability of silicone and Laminac 4231 cores exposed to 100 percent relative humidity at 100°F. for 21 days. The strength tests show that Laminac 4231 cores are much stronger than silicone cores of the same density at temperatures below 300°F. Above 300°F., the strength of Laminac 4231 cores approaches the strength of silicone cores. The weight and dimensional ability of silicone cores were not affected by exposure to high humidity. Laminac 4231 cores showed increases in both weight and dimension after the high-humidity exposure.

October 1956

SUBJECT: DEVELOPMENT AND EVALUATION OF THE CLIMBING PEEL METHOD FOR TESTING ADHESIVE BONDS IN SANDWICH AND METAL-TO-METAL CONSTRUCTIONS

INVESTIGATOR: H. W. Eickner  
Fred Warren

CONTRACT: AF 33(616)-56-9

CONTRACTOR: Forest Products Laboratory

ABSTRACT: A method of test was developed for determining the peel strength of adhesive bonds in sandwich and metal-to-metal constructions. This method of test, called the FPL climbing peel test, was compared with several other peel test methods. Test data were obtained on matched specimens from sandwich panels of aluminum faces bonded to aluminum honeycomb cores and from bonded aluminum-to-aluminum panels. Eleven adhesive processes were used in preparing the test panels.

All the peel tests investigated appeared to rank the peel resistance of the different adhesive bonds in approximately the same order. The climbing peel test is recommended as a standard test method, however, because of its simplicity, uniformity of peeling, and its adaptability to tests at other than room temperature and to tests of both sandwich and metal-to-metal constructions.

April 1957

SUBJECT: MECHANICAL PROPERTIES OF 422-J BACFOAM CORE FOR SANDWICH CONSTRUCTION

INVESTIGATOR: P. M. Jenkinson  
E. W. Kuenzi

CONTRACT: DO 33(616)-56-9

CONTRACTOR: Forest Products Laboratory

ABSTRACT: This report presents the results of a few edgewise compression and flexure tests conducted at the U. S. Forest Products Laboratory on sandwich constructions of Boeing 422 J Bacfoam core with a density of 17 to 20 pounds per cubic foot and 2024-T3 clad aluminum facings 0.040 inch thick, and the results of flatwise compression tests on the core. Tests were conducted at room temperature, 200°, 300°, 400°, and 500°F. The test specimens and methods of testing are described. Test data are presented in tabular form and as curves illustrating the variation of mechanical properties with temperature. Typical stress-strain curves are included for flatwise compression of core at room temperature.

Strength properties of the core depended on the size of the core air voids. Coarse-textured core was about 60 percent as strong as fine-textured core in flatwise compression, but coarse-textured core was slightly stronger in shear. Flatwise compression and shear strength values of the core at 500°F. were 40 to 50 percent of the values at room temperature. The shear modulus at 500°F. was about 25 percent of that at 75°F. The edgewise compressive strength of sandwich at 500°F. was about 60 percent of the strength at room temperature.

## TESTS, NONDESTRUCTIVE

### TEXTILES

WADC TR 54-612

October 1956

ASTIA Document No. AD 110419

OTS Release

SUBJECT: DEVELOPMENT OF DYEING FORMULATIONS FOR WOOL/SYNTHETIC BLENDS  
FOR USAF SHADE BLUE 84

INVESTIGATOR: Robert J. Peirent  
Adolph Katz

CONTRACT: AF 33(600)-16396

CONTRACTOR: Lowell Technological Institute Research Foundation

ABSTRACT: Dyeing formulae were developed for viscose, nylon, Dacron, Dynel, Acrilan and Orlon fibers to obtain suitable Blue Shade #84 wool/synthetic fabrics for U. S. Air Force uniforms. At the time that the development phase was terminated, no formulae were found which would give the desired colorfastness properties for Dynel and Acrilan. Consequently, these fibers were eliminated from the production phase of the project. (Subsequent developments have demonstrated that new techniques will afford adequate fastness on these fibers. However, the project had progressed to a point which precluded reconsideration of these fibers.)

A 100% wool control fabric and a series of wool/synthetic fabrics containing 10, 20, and 30% synthetic fiber were manufactured for subsequent evaluation of their physical and colorfastness properties. These fabrics were made to conform to the specifications set forth in MIL-C-849, Cloth, Wool, Serge, Blue Shade 84.

A comprehensive study of the properties of the wool and the wool/synthetic fabrics reveals that they meet the physical requirements desired and have adequate colorfastness as well as high resistance to fading.

The scope of this study was too broad to permit examination of all of the dyestuffs produced domestically or at all of the possible dyestuff combinations which conceivably would meet target properties; however, as many dyes and formulae were included as possible. Due to the rigid requirements of this investigation, many dyes and formulae were evaluated in conditions for which they were not intended. Hence, it must not be assumed that the results tabulated herein are equally valid for other test conditions or applications, nor is it to be construed that a dye or formula is not entirely satisfactory for the manufacturers intended use or advertised claims. Further, it is not to be construed that formulae other than those covered in this report cannot perform equally satisfactorily. The disclosure of dye formulae, dyeing procedures, and methods of colorimetry described herein does not constitute license for practice. The selection of a particular dye formula for producing fabric required in this project does not imply approval of the U. S. Air Force for the specific dye or formula for producing Blue Shade 84.

WADC TR 55-135

September 1956

ASTIA Document No. AD 97241

SUBJECT: DEVELOPMENT OF DACRON PARACHUTE MATERIALS

INVESTIGATOR: Ernest R. Kaswell

Myron J. Coplan

CONTRACT: AF 33(600)-24087

CONTRACTOR: Fabric Research Laboratories, Inc.

ABSTRACT: Certain Air Force deceleration parachutes are exposed to temperatures in the 350-400°F. range. Nylon is severely degraded when exposed to these temperatures for relatively short time periods. Dacron exhibits significantly better heat degradation resistance. Selected threads, braids, cloths, webbings, ribbons and tapes composed of Dacron rather than nylon were designed, developed and delivered to Wright Air Development Center. All confirm the improved heat degradation resistance.

Nylon is widely used in parachutes because of its high tenacity (strength/weight ratio) and energy absorption. Dacron's tenacity and energy absorption are slightly lower. However, after heat exposure for 24 hours at 350°F. these values remain far higher than for nylon. While Dacron does not degrade at high temperatures, it does shrink appreciably. This required that all Dacron items be stabilized via heat relaxation shrinkage at 350-375°F. The additional shrinkage produced elongations of about twice the order of those currently found in nylon. Furthermore, this shrinkage increased the weight/length or weight/area of the Dacron items, and thus, the strength/weight ratio was further diminished as compared with nylon.



Nylon is outstanding in its ability to deform under load and recover upon load removal without evidencing a large amount of non-recoverable elongation or permanent set. Dacron, in its originally produced state has almost as good recovery properties. However, the shrinkage incorporated into Dacron via heat relaxation manifests itself as non-recoverable elongation upon load application. Thus, at about 90% of rupture load, permanent set amounts to about two-thirds of the total elongation present in the shrunk Dacron items. At lower stress levels the amount of permanent set diminishes and the recoverable elongation increases.

It has been determined that losses in strength, elongation and energy of the subject Dacron materials after heat ageing for 24 hours at 350°F amount to about 10-20%.

A significant increase in stiffness of the Dacron items occurs upon heat ageing, and this was definitely ascribed to the finish applied by the Dacron filament producer. Such finishes can be removed by simple detergent scouring.

In conjunction with the development of heat stable Dacron yarns, preliminary studies indicate that a proper sequence of stretching and relaxing at below-rupture loads and at high temperatures, will produce Dacron yarn of high tenacity, normal elongation and zero heat shrinkage.

WADC TR 55-297  
ASTIA Document No. AD 97242  
OTS Release

September 1956

SUBJECT: DEVELOPMENT OF HIGH TENACITY-HEAT STABLE DACRON YARNS  
INVESTIGATOR: Robert J. Coskren  
Thomas T. Constantine  
CONTRACT: AF 33(600)-24087  
CONTRACTOR: Fabric Research Laboratories, Inc.  
ABSTRACT: Dacron yarn, because of its superior resistance to thermal degradation when exposed to temperatures of 350-400°F. for prolonged periods, has been suggested as a replacement for nylon in deceleration parachutes. Dacron's strength retention after high temperature exposure is good, but a longitudinal shrinkage of the order of 20% takes place which presents problems of parachute component dimensional stability. Secondly, this 20% shrinkage is reflected in lower strength and energy to weight ratios, thus requiring proportionately heavier parachutes. Furthermore, the added elongation resulting from thermal shrinkage is composed primarily of secondary creep or permanent set. Upon deployment of the parachute the possibility exists that fabric components might deform at the time of stress application, but not recover upon stress removal unless and until the parachute or its components are again elevated to the 350°F level.

At the inception of this phase of the study there was available from the duPont Company high tenacity (6.1 grams/denier), nominal rupture elongation (9.2%), high thermal shrinkage (20%) Dacron yarn. By free relaxation at an elevated temperature this yarn could be converted to medium tenacity (4.8 grams/denier), high elongation (36%), low shrinkage ( $<2\%$ ) yarn.

Preliminary experimentation showed that cyclical yarn stressing and relaxing processes at elevated temperatures would produce a yarn of the desired high tenacity, nominal rupture elongation and low shrinkage, provided that the yarn was allowed to relax completely after the last stressing cycle.

In an attempt to develop Dacron yarn of optimum properties, those factors which were found to have an influence on ultimate properties, were thoroughly investigated. These included stretching temperatures, times, and amounts and sequences of stretching-relaxing systems.

The optimum process so far developed consists of three basic steps, namely:

1. 20% hot stretch at yarn temperatures of 340-390°F.
2. Fixed length at 430-450°F.
3. Free shrinkage at 350°F.

Yarn produced by such a process has a tenacity of 6.7-7.0 grams/denier, an elongation of 14-16% and a shrinkage of less than 2% at 350°F.

This process is an improvement over the multiple cycle methods originally studied. Furthermore, it is undoubtedly cheaper in terms of both equipment cost and production.

Concomitant with the research, a pilot processing machine was developed and some consideration was given to the commercial practicability of the process insofar as production speed was concerned.

It is recommended that this study be continued in order to further determine those factors which influence the development of an optimum yarn. Also, the ultimate design and construction of a commercial prototype machine is needed in order to produce yarn in sufficient quantities so that end items may be prepared and thoroughly evaluated.

July 1956

SUBJECT: RESEARCH AND DEVELOPMENT OF ABRASION RESISTANT TREATMENTS  
FOR DACRON WEBBINGS

INVESTIGATOR: George Thomson  
Joseph S. Panto  
Ernest R. Kaswell

CONTRACT: AF 33(616)-2563

CONTRACTOR: Fabric Research Laboratories, Inc.

ABSTRACT: The purpose of the work herein reported was the development of finishes which could be applied to Dacron webbing with resulting increase in abrasion resistance. The finish was to remain flexible at -65°F. and be stable to artificial sunlight for 100 hours and to a temperature of 350°F. for 16 hours.

A satisfactory method for determining flexibility of webbings at standard conditions and at -65°F. has been developed.

Preference was given to commercially available water dispersions of a number of different types of resins such as acrylic, acrylonitrile, natural rubber and silicones because of their freedom from hazards of toxicity and flammability and their ease of handling.

Webbings treated with one particular silicone and catalyst emulsion were superior to all other treated samples from the point of view of abrasion resistance, low temperature flexibility and resistance to heat aging. However, the resistance to artificial sunlight for 100 hours was lowered so that the webbing retained only 60 to 70% of the strength of a similarly exposed untreated sample, whereas a 90% retention was specified. In spite of this, the other aforementioned properties were so superior that it was decided to commercially apply this silicone treatment to 250 yards of Dacron webbing.

September 1956

SUBJECT: A STUDY OF THE EFFECTS OF CHEMICALS ON THE PROPERTIES OF  
PARACHUTE FABRICS

INVESTIGATOR: J. Glenn Templeton

CONTRACT: AF 33(616)-2530

CONTRACTOR: School of Textiles North Carolina State College

**ABSTRACT:** During routine inspection of parachutes, several badly degraded nylon parachutes were discovered. Preliminary evaluation by chemical analysis indicated the presence of mineral acids. To provide background data, nylon and Dacron fabrics were exposed to sulfuric, hydrochloric, nitric, phosphoric, sulfurous, hydrosulfuric, and nitrous acids. The exposures were conducted under various concentrations for periods of time up to six months. The evaluation was conducted by establishing the breaking strength of the exposed fabrics and comparing them to the original breaking strength.

Additional phases of the work consisted of studying the effect of light in combination with the acids, studying the effect of sulfur dioxide, hydrogen sulfide, and oxides of nitrogen, and examinations of procedures for detecting, by visual means, degradation by acids.

Dacron was found to be quite resistant to attack by mineral acids in comparison with nylon. Acid-treated nylon was more sensitive to light than acid-treated Dacron.

WADC TR 55-340 Sup 1  
ASTIA Document No. AD 110524  
OTS Release

November 1956

**SUBJECT:** A STUDY OF THE EFFECTS OF CHEMICALS ON THE PROPERTIES OF PARACHUTE FABRICS  
**INVESTIGATOR:** David M. Cates  
**CONTRACT:** AF 33(616)-2530  
**CONTRACTOR:** School of Textiles North Carolina State College  
**ABSTRACT:** The results given in the Technical Report WADC TR 55-340 were obtained by determining the percent loss in strength of nylon and Dacron parachute fabrics when the fabric was treated with chemicals under various conditions. Because fabrics are not absolutely uniform and experimental procedures are not perfectly reproducible, the results of breaking strength tests varied. Since each sample consisted of several breaking tests (usually 10), it was possible to estimate the within sample reliability by statistical means. This was done by calculating the 95% confidence limits.

WADC TR 55-379  
OTS Release

May 1956

**SUBJECT:** THE EFFECT OF FIVE SYNTHETIC LUBRICANTS ON USAF FABRICS  
**INVESTIGATOR:** Richard A. Sublette, 1/Lt

**ABSTRACT:** This investigation was conducted to determine if textile fibers and fabrics employed by the USAF are subject to deterioration or degradation when exposed to synthetic lubricants. In order to accomplish the desired program, two series of fabrics, composed of fibers that are commonly used in USAF fabrics, were prepared by immersing one series in a similar group of lubricants at 160°F. Both series were immersed in the lubricants for 72 hours. After exposure, laundering and dry cleaning tests were conducted on the fabrics. Diaphragm burst strength tests indicated the synthetic lubricants did not cause a loss in strength of the fabrics.

WADC TR 55-465  
OTS Release

May 1956

**SUBJECT:** THE DEVELOPMENT OF HIGH STRENGTH NYLON PARACHUTE FABRICS

**INVESTIGATOR:** Hamilton J. Bickford

Thomas L. Rusk, Jr.

Donald K. Kuehl

**CONTRACT:** AF 33(600)-29135

**CONTRACTOR:** Cheney Brothers

**ABSTRACT:** Seven nylon fabrics were developed that should give stability to dry air for use at temperatures up to 250°F for at least 16 hours with little or no significant difference in effect between continuous or intermittent exposure. Severe degradation takes place when exposed to temperatures of 350°F.

Permeability and strength requirements were met and certain information on the relationship between permeabilities at different pressure levels developed.

Seam efficiency test methods and breaking strength test methods at strengths over 500 lb/in could be improved.

Nylon fabrics can be processed to provide a balanced fabric (coinciding warp and filling load-elongation curves within 15%) for the lower breaking strength fabrics.

WADC TR 55-485  
OTS Release

May 1956

**SUBJECT:** AN APPARATUS FOR DETERMINING BIAxIAL STRENGTH PROPERTIES OF CLOTH AND SUPPORTING TEST DATA

**INVESTIGATOR:** Winston C. Boteller

**CONTRACT:** AF 33(616)-2857

**CONTRACTOR:** Georgia Institute of Technology

**ABSTRACT:** The Georgia Tech biaxial fabric tension testing machine was modified to permit the simultaneous recording of warp and filling load-elongation curves. It was the purpose of these studies to determine the elastic properties of selected nylon, Orlon, and Dacron parachute-type cloth under various conditions of biaxial loading.

Tests conducted at various speeds to determine the effect of testing speed on elastic properties indicated that the testing speed has no apparent effect on the elastic properties. A comparison of elongation measurements by extensometer and jaw separation indicates that jaw separation measurements are satisfactory for light loads, but the difficulty of securely clamping the sample introduces serious discrepancies at high loads. The measurement of elongation at various locations in the sample shows a slight increase in elongation as the extensometer is moved towards the clamping jaws.

WADC TR 55-494  
OTS Release

May 1956

**SUBJECT:** THE DEVELOPMENT OF CURRENT NYLON WEBBINGS UTILIZING 840 DENIER YARNS IN LIEU OF NOW SPECIFIED 210 DENIER YARNS

**INVESTIGATOR:** Russell J. Neff

**CONTRACT:** AF 33(600)-29034

**CONTRACTOR:** Phoenix Trimming Company

**ABSTRACT:** A series of nylon webbings was developed by the Phoenix Trimming Co. using 840 denier yarns in lieu of the presently used 210 denier yarns. A second series of webbings was manufactured using 210 denier yarns as required by Specification MIL-W-4088B. Comparative tests were conducted between these two constructions to determine whether the use of 840 denier yarns was feasible. In addition, four webbings were manufactured in accordance with Specification MIL-W-5625 except that a solid weave was used in lieu of tubular. These webbings were also manufactured using both types of yarn and a series of comparative tests were conducted. Five experimental high tensile strength webbings were manufactured using 840 denier yarns only. These experimental webbings were subjected to similar tests. All of the webbings were piece dyed except the webbings manufactured in accordance with Specification MIL-W-5625 and the 1" 14000 pound, 1 3/4" 25000 pound, and 3" 35000 pound experimental webbings.

This investigation has shown that 840 denier yarn appears as good and sometimes better than 210 denier yarn. Also, results have indicated that piece dyeing of the webbing is not only feasible, but desirable from the standpoints of giving better original tensile strength and abrasion resistance.

June 1956

SUBJECT: RESEARCH AND DEVELOPMENT OF ABRASION RESISTANT TREATMENTS  
FOR NYLON WEBBINGS

INVESTIGATOR: George Thomson Myron J. Coplan  
Joseph S. Panto Ernest R. Kaswell

CONTRACT: AF 33(616)-2703

CONTRACTOR: Fabric Research Laboratories, Inc.

ABSTRACT: The purpose of the work herein reported was the development of finishes which could be applied to nylon webbings with resulting increase in abrasion resistance. The finish was to remain flexible at -65°F. and be stable to artificial sunlight for 100 hours and to a temperature of 170°F. for 16 hours.

The method employed for measuring flexibility was the same as that developed under a similar investigation on Decron Webbing as described in WADC TR 55-313.

Preference was given to commercially available water dispersions of a number of different types of resins such as acrylic, acrylonitrile, natural rubber and silicones because of their freedom from hazards of toxicity and flammability, and their ease of handling.

Webbings treated with one particular silicone and catalyst emulsion were superior to all other treated samples from the point of view of abrasion resistance, low temperature flexibility and resistant to heat ageing. The application of this silicone finish did not materially affect the resistance of the nylon webbing to 190 hours exposure to artificial sunlight. This finish was consequently applied to 194 yards of Type VIII O.D. and 478 yards of Type X O.D. nylon webbing.

WADC TR 56-256  
ASTIA Document No. AD 118227  
OTS Release

April 1957

SUBJECT: EVALUATION OF DUPONT AND CHEMSTRAND NYLON YARN

INVESTIGATOR: Peter Y. Stanton, 1/Lt

ABSTRACT: The investigation undertaken herein was two phase, namely:

1. To compare nylon yarns of various deniers manufactured by the E. I. duPont de Nemours and Company with nylon yarns of the same denier manufactured by the Chemstrand Corporation.

2. The service testing of personnel parachutes of which the canopy material was fabricated from Chemstrand nylon and the remainder of the parachute fabricated from duPont nylon as a comparison to the same type parachutes fabricated wholly from duPont nylon.

A comparison of the nylon yarns manufactured by duPont with the nylon yarns manufactured by Chemstrand shows no meaningful difference between the two with regard to breaking strength, tenacity, elongation, energy absorption and melting point. Also, a comparison of the high and low temperature characteristics of the yarns manufactured by both companies shows no real difference between the two with regard to the properties investigated.

Service tests have shown that the parachutes fabricated from Chemstrand yarn in the canopy are equal in performance to those manufactured wholly from duPont yarn.

WADC TR 56-257  
ASTIA Document No. AD 110570  
OTS Release

November 1956

SUBJECT: DEVELOPMENT OF STATIC LINE WEBBING FOR THE T-10 PARACHUTE SYSTEM

INVESTIGATOR: Peter Y. Stanton, 1/Lt.

ABSTRACT: The purpose of this program was the development of static line webbing for the T-10 parachute system. The objective of this development is to replace the presently used inferior static line webbing with webbing having more satisfactory qualities. A silk webbing and linen webbing were developed along with six nylon webbings each of which had a different elongation. Subsequent to their development the webbings were forwarded to the Parachute Branch, Equipment Laboratory, for further evaluation.

The use of silk in static line webbing does not appear feasible because of the unavailability of raw silk. The use of linen in static line webbing was unsatisfactory because of the low elongation, low energy absorption qualities of linen. The developed nylon webbings appear satisfactory for use as static line webbing.

WADC TR 56-288  
ASTIA Document No. AD 110558  
OTS Release

November 1956

SUBJECT: A STUDY OF THE EFFECTS OF CHEMICALS ON THE STRENGTHS OF NYLON AND DACRON PARACHUTE FABRICS

INVESTIGATOR: David M. Cates



CONTRACT: AF 33(616)-2891  
CONTRACTOR: School of Textiles North Carolina State College  
ABSTRACT: The effect of certain organic solvents and solutions of inorganic substances on the strength of nylon and Dacron parachute fabrics was investigated. The fabrics were exposed to each of the reagents and then aged under different conditions for a period of six months. Breaking strength tests were made on the fabrics at monthly intervals. For each testing condition, the concentration of reagent, ageing temperature, and ageing condition were varied. Ageing was carried out under three kinds of conditions: namely, with the fabrics immersed in solution; with the fabrics first immersed in the solution, then extracted and aged under low humidity conditions; with the fabrics first immersed in solution, then extracted and aged under high humidity conditions.

It was found that in general, an increase in concentration, temperature, or time gave higher strength losses of both nylon and Dacron fabrics. In most instances the same trend in loss of strength was observed whether the fabrics were aged in solution, at high relative humidity, or at low relative humidity. Finally, the data showed that the reagents could generally be ranked in three groups according to the effect produced in the fabric: (1) reagents causing small strength losses even at high concentrations and high temperature; (2) reagents causing large strength losses at high concentrations and high temperatures; and (3) reagents causing large strength losses even at low concentrations and low temperature.

WADC TR 56-541  
ASTIA Document No. AD 118229  
OTS Release

April 1957

SUBJECT: EFFECTS OF FUNGISTATS ON PARACHUTE SUSPENSION LINE LUBRICANT PERFORMANCE  
INVESTIGATOR: Theodore L. Eriksson  
CONTRACT: AF 33(616)-3311  
CONTRACTOR: Wyandotte Chemicals Corporation  
ABSTRACT: Nylon parachute canopy cloth is subject to occasional failure due to "line burns" produced by sliding of the suspension lines over the canopy during opening. As a means of reducing these failures, ten candidate lubricants were selected by Lowell Technological Research Foundation under Contract No. AF 18(600)-136, (WADC TR 54-323). These lubricants were, however, found subject to mildew attack, and the present work was undertaken with the object of determining their lubricating properties on addition of a maximum of 2% of each of three specified mildew inhibitors.

A maximum lubricant loading of approximately 10% by weight of the suspension line was desirable. Values of  $9.5 \pm 0.5\%$  were obtained with all inhibitor combinations of nine of the ten lubricants by deposition from

homogeneous trichlorethylene solutions. One lubricant consisted of emulsified lubricant in water.

Mildew inhibition tests were performed according to a specific procedure developed at WADC and resulted in the elimination of all but two candidate combinations. These candidate combinations showed no decrease in lubricity from the uninhibited lubricants when subjected to "friction fusion" tests on apparatus developed under Contract AF 18(600)-136, and little or no decrease in lubricity after oven ageing.

Breaking strength tests performed by the Materials Laboratory, WADC, showed that one of the two combinations was detrimental to the breaking strength of the suspension line when subject to oven ageing. The surviving combination consisted of Spermaceti wax plus 2% dialkyldimethylammonium bromide (Isothan DL-1).

### TRANSPARENT MATERIALS

WADC TR 53-378 Pt 3

April 1956

SUBJECT: THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET  
INVESTIGATOR: George A. Dalin  
Joseph Rennert  
CONTRACT: AF 33(616)-111  
CONTRACTOR: Balco Research Laboratories  
ABSTRACT: The deposition of transparent electrically conductive films on acrylic sheet for the purpose of eliminating fog and ice formation by dissipation of electrical power is discussed. Techniques are described for forming such films by sputtering. The optical, mechanical and electrical properties are described. Suggestions for the design of transparent sections are presented.

WADC TR 55-499

April 1956

SUBJECT: AN INVESTIGATION OF THE RAIN EROSION OF TRANSPARENT AIRCRAFT GLAZING MATERIALS AT SUBSONIC SPEED  
INVESTIGATOR: Roy R. Lapp  
Raymond H. Stutzman  
Norman E. Wahl  
CONTRACT: AF 33(600)-6469  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

**ABSTRACT:** The rain erosion resistance of glass and transparent plastic materials currently used for glazing was determined at 500 mph and 1 in/hr rainfall on the Cornell Aeronautical Laboratory test apparatus.

Three glasses with coefficients of linear thermal expansion ranging from 30 to  $90 \times 10^{-7}$  in/in/°C were evaluated. Each glass was tested in the annealed, semi-tempered and fully tempered state.

The fully tempered hard lime glass possessed the greatest rain erosion resistance.

The transparent plastic materials tested were Plexiglas II UVA, Plexiglas 55, Polymer K, Sierracin and polymethyl-alpha-chloroacrylate.

In these tests all the transparent plastic materials eroded in one to three minutes. Similar specimens in glass withstood erosion 10 to 15 times as long as the transparent plastic materials, i.e. they showed erosion in from ten to thirty minutes. In general, the erosion resistance of the same type of glass increased as the temper of the glass was increased.

It might be stated that aircraft glazing materials have shown little tendency to pit due to rain erosion in service. This is due to the fact that angle of impact of rain drops upon the glazing material in flight attitude is low. In the tests described herein, the impact angle is essentially 90° which results in the highest rate of erosion.

WADC TR 56-133  
ASTIA Document No. AD 97191

August 1956

**SUBJECT:** RESISTANCE TO SHATTERING BY GUNFIRE OF TRANSPARENT  
LAMINATED MATERIALS FOR AIRCRAFT GLAZING

**INVESTIGATOR:** Edward M. Petruska

**CONTRACT:** AF 33(616)-2479

**CONTRACTOR:** Rohm & Haas Company

**ABSTRACT:** Gunfire tests were conducted on laminated Plexiglas 55, Sierracin 611 and Polymer K with a 300°F outside surface temperature, a 70°F inside air temperature, and a 4 psi differential on each specimen. The specimens were flat and clamped around the periphery. The tests were run with a 50 caliber bullet fired at each test sample.

When comparing laminates of equal configuration, Plexiglas 55 had better crack resistance than Polymer K and both were better than Sierracin 611. Orientation of laminates of non-uniform configuration had little effect on crack resistance. The 0.375"-0.375" Polymer K laminate was slightly better than other Polymer K configurations tested. The 0.400"-0.200"-0.400" Sierracin 611 laminate was much better than any other Sierracin configuration tested. Laminates containing 25 parts DBS plasticizer per 100 parts PVB were slightly better than those containing 37.5 parts plasticizer.

Some 75% stretched Plexiglas 55 of .250" thickness was tested under the same conditions as the laminates. These resulted in a bullet hole with no cracking. The stretched material was therefore superior in shatter resistance to all the laminates tested.

WADC TR 56-159  
ASTIA Document No. AD 97139

July 1956

SUBJECT: SILICONE INTERLAYER MATERIAL PROGRAM

INVESTIGATOR: Keith E. Polmanteer  
Francis J. Campbell  
Thomas L. Laur

CONTRACT: AF 33(600)-27185

CONTRACTOR: Dow Corning Corporation

ABSTRACT: The further development of a thermally stable silicone interlayer material for laminated glass, originally introduced under AF Contract 33(600)-23081 and continued under AF Contract 33(600)-27185, was the object of this research by the Dow Corning Corporation during a one year period ending in January, 1956. Improvements were made in fabricating and laminating the interlayer materials. Two new interlayer materials, Type K and Type L, were described. Both of these materials gave much better adhesion to glass and to aluminum than did the previous silicone interlayer materials studied. The materials both look promising for use in aircraft high temperature applications. Glass laminates made with Type K interlayer material will withstand temperatures up to 300°F for an indefinite period of time. Although Type L interlayer material requires further minor improvements, it appears to be thermally stable at temperatures of 400° to 450°F.

WADC TR 56-243  
ASTIA Document No. AD 118035

February 1957

SUBJECT: MODIFIED POLYMETHYL ALPHA-CHLOROACRYLATE PLASTIC SHEET

INVESTIGATOR: Harry D. Anson

CONTRACT: AF 33(600)-23883

CONTRACTOR: General Aniline and Film Corporation

ABSTRACT: This program was initiated to obtain a transparent polymer with elevated temperature properties superior to polymethyl  $\alpha$ -chloroacrylate. A modified polymethyl  $\alpha$ -chloroacrylate was developed with markedly improved resistance to bubbling (chemical heat stability), but with no or only a slight increase in physical strength as measured by the heat distortion test. The modified polymer exhibited a marked increase in the already excellent resistance to crazing of polymethyl  $\alpha$ -chloroacrylate, but the modified polymer possessed a decreased light stability. Although the modified polymer is less formable than polymethyl  $\alpha$ -chloroacrylate, the formability is still quite good.

In addition to the improved chemical heat stability and improved resistance to crazing, the modified methyl  $\alpha$ -chloroacrylate polymer was found to exhibit the property of being obtainable with better polished surfaces after annealing than polymethyl  $\alpha$ -chloroacrylate cast under the same conditions.

WADC TR 56-352  
ASTIA Document No. AD 118248

April 1957

SUBJECT: DEVELOPMENT OF A RIGID TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURE  
INVESTIGATOR: George Helvey  
H. A. Clark  
CONTRACT: AF 33(616)-3065  
CONTRACTOR: Dow Corning Corporation  
ABSTRACT: This program was initiated in an attempt to obtain a rigid, transparent plastic having properties at elevated temperatures superior to those of presently available transparent plastic materials.

Evaluation of several silicone formulations was conducted. The most promising material was a combination of high and low viscosity components, designated as Resin Type EB.

This Resin Type can be polymerized in cast sheet form and is thermally stable after prolonged exposure at 200°C. Optical properties of the cast sheet are excellent and not visibly affected by heat aging. The physical properties are, in general, well below those of known materials (Heat Distortion is 40°C to 66°C).

WADC TR 56-491  
ASTIA Document No. AD 110619

December 1956

SUBJECT: DETERMINATION OF THE ACCEPTABILITY OF A COMMERCIAL STRETCH-FORMED ACRYLIC AIRCRAFT CANOPY  
INVESTIGATOR: David M. Coddington, 1/Lt  
ABSTRACT: The first commercially stretch-formed acrylic aircraft canopy was tested for adequate crack propagation resistance and edge attachment strength, stretch distribution and birefringence. Results indicate crack propagation resistance values of 25-36 in.lbs/in<sup>2</sup> and edge attachment strength well above the 600 lbs/in. design requirement.

Based on the results of this evaluation, the canopy is considered satisfactory.

March 1957

SUBJECT: EFFECTS OF GAMMA RADIATION ON AIRCRAFT TRANSPARENT MATERIALS  
INVESTIGATOR: Donald L. Schmidt, 1/Lt  
Robert H. Johnson, 1/Lt

ABSTRACT: The effects of gamma radiation on various optical, mechanical and thermal properties of transparent plastic materials were investigated. Gafite, Plexiglas 55 and Sierracin 611 materials were irradiated in a gamma facility to total absorbed doses of  $10^6$  to  $10^{10}$  ergs per gram and then evaluated in accordance with standard testing procedures.

The physical properties of the transparent plastics were not significantly affected by a radiation exposure of  $10^7$  ergs per gram, or less. At higher absorbed doses, most of the physical properties were degraded. In addition, several desirable changes in specific physical properties were also obtained.

The optical properties of the transparent plastic materials were more susceptible to radiation-induced changes than were the mechanical or thermal properties.

WADC TR 56-645 Pt I  
ASTIA Document No. AD 118323

May 1957

SUBJECT: PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES Part I  
Preliminary Program  
INVESTIGATOR: Matthew J. Kerper  
Campbell Lathey  
Henry E. Robinson  
CONTRACT: AF 33(616)-56-13  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: A program has been initiated to investigate the physical

properties of glasses that are suitable for aircraft glazing. The program will consist of: 1) developing suitable test methods for determining the physical properties at room and elevated temperatures, and 2) determining the values of the physical properties of individual glasses for use as design data.

During the past year all necessary test methods have been developed or adopted. A satisfactory modulus-of-rupture test was developed using two-point loading and specimens that had one surface abraded by sandblasting. The sandblasting reduced the spread of the results and made the test more sensitive.

Some thermal properties previously determined have been adopted and some mechanical properties for a commercial soda lime glass have been determined at room and elevated temperatures.

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